N66-15186

NASA CR-54718

# VON KARMAN CENTER

SNAP-8 DIVISION

## SNAP-8 MATERIALS REPORT FOR JULY-DECEMBER 1964

BY H. DEROW AND B. E. FARWELL

CONTRACT NAS 5-417

A REPORT TO

NATIONAL AERONAUTICS AND SPACE ADMINISTRATION
LEWIS RESEARCH CENTER

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Contract NAS 5-417

A Report To

NATIONAL AERONAUTICS AND SPACE ADMINISTRATION
LEWIS RESEARCH CENTER
SNAP-8 PROJECT OFFICE
H. O. SLONE, SNAP-8 PROJECT MANAGER

Report No. 2989 January 1965

AEROJET-GENERAL CORPORATION
A SUBSIDIARY OF THE GENERAL TIRE & RUBBER COMPANY

# FOREWORD

Aerojet-General Corporation is proceeding with the design and development of the SNAP-8 Power Conversion System, as authorized by National Aeronautics and Space Administration Contract No. NAS 5-417.

The ultimate objective of the SNAP-8 Program is to design and develop a 35-kw electrical generating system for use in various space missions. The power source will be a nuclear reactor furnished by the Atomic Energy Commission. The SNAP-8 system will use a eutectic mixture of sodium and potassium (NaK) as the reactor coolant and will operate on a Rankine cycle, with mercury as the working fluid for the turbogenerator. The SNAP-8 system will be launched from a ground base and will be capable of unattended full-power operation for a minimum of 10,000 hours. After the system is placed into orbit, activation and shutdown may be accomplished by ground command.

This semiannual materials report is submitted in partial fulfillment of the contract. Part of the information appearing in this volume was prepared at Aerojet-General Nucleonics, San Ramon, California, under Aerojet-General Corporation Subcontract 274949.

The Component Materials Development Program was under the management of R. S. Carey, Head, Technical Support Dept., SNAP-8 Division, Von Karman Center. The work at the Von Karman Center was done under the direction of H. Derow, Head, Materials Section, SNAP-8 Division. The work at Aerojet-General Nucleonics was done under the direction of B. E. Farwell, Head, SNAP-8 Section, Metallurgy Dept., Applied Science Division. The following engineers contributed to the various programs:

At Von Karman Center - H. Bleil, J. Barry, F. Cassidy, C. Landusky and R. McPherson.

At Aerojet-General Nucleonics - A. R. Herdt, B. Kountanis, E. F. McDaniel, J. H. Ralphs, and M. K. Wong.

The work performed by R. Bear of the NASA Lewis Research Center in evaluating the refractory bimetal tube material diffusion specimens is gratefully acknowledged.

The suggestions and guidance of W. Titus and M. Parkman, Metallurgy Department, Aerojet-General Nucleonics; and P. Stone and J. Merutka, SNAP-8 Project Office, Lewis Research Center, NASA, are gratefully acknowledged.

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## GLOSSARY

Abbreviations commonly used in the SNAP-8 Program are defined below.

| | |
|---|---|
| AA | Alternator assembly |
| AEC | Atomic Energy Commission |
| AGC | Aerojet-General Corporation |
| AGN | Aerojet-General Nucleonics |
| AI | Atomics International |
| AOC | Award of contract |
| ATL | Acceptance test loop |
| AZFO | NASA-Azusa Field Office |
| BOD | Beneficial occupancy date |
| CGEST | Cold-gas electrical system test |
| CL | Corrosion loop (AGN) |
| CPC | Ceramic potting compounds |
| CTL | Component test loop (AGN) |
| DDAS | Digital data acquisition system |
| DWG | Drawing |
| EDM | Electrical-discharge machining |
| EFF | Efficiency |
| EGS | Electrical generating system |
| EM | Electromagnetic |
| EME | Electromagnetic equivalent |
| FPS | Flight prototype system |
| FPTF | Flight prototype test facility |
| FRA | Flight radiator assembly |
| GE | General Electric Company |
| GN$_2$S | Gaseous nitrogen stand |
| GPS | Ground prototype system |
| GPTF | Ground prototype test facility |
| HML | Heavy coating of ML (q.v.) |
| HR | Heat rejection |
| HRF | Heat rejection fluid |
| HRL | Heat rejection loop |
| HRS | Heat rejection system |
| HTL | Heat-transfer loop |
| L/C | Lubricant/coolant |
| L/CL | Lubricant/coolant loop |
| LeRC | Lewis Research Center |
| LML | Liquid mercury loop |
| LMS | Liquid mercury stand |
| LNL | Liquid NaK loop |
| LOL | Liquid organic loop |
| LOS | Liquid organic stand |
| LPL | Low power loop |
| MIS | Mercury injection system |
| ML | Pyre-ML, Du Pont polyimide organic resin; as employed in statorette serial numbers, indicates the use of this substance |
| MLA | Mercury loop assembly |
| MN$_2$S | Mercury-nitrogen system |
| MPMA | Mercury pump motor assembly |
| NaK | Eutectic mixture of sodium and potassium |
| NASA | National Aeronautics and Space Administration |
| NF | Nuclear facility |
| NHRA | NaK heat-rejection assembly |
| NPA | NaK pump assembly |

## GLOSSARY (cont.)

| | |
|---|---|
| NPMA | NaK pump motor assembly |
| NPS | Nuclear power system |
| NPSH | Net positive suction head |
| NS | Nuclear system |
| NSL | NaK simulation loop |
| ORNL | Oak Ridge National Laboratory |
| PBRF | Plum Brook Reactor Facility |
| PCS-1 | Power Conversion System No. 1 |
| PCS-2 | Power Conversion System No. 2 |
| PCS-3 | Power Conversion System No. 3 |
| PCS-4 | Power Conversion System No. 4 |
| PF | Power factor |
| PL | Primary loop |
| PLR | Parasitic load resistor |
| PMA | Pump motor assembly |
| PNLA | Primary NaK loop assembly |
| PO | Purchase order |
| PTAT | Preliminary type-approval test |
| PVT | Pressure-volume-temperature |
| $R_B$ | Rockwell B (hardness) |
| RPL | Rated power loop |
| SC | Speed control |
| S8DS | SNAP-8 development system |
| S8ER | SNAP-8 experimental reactor |
| SL-1 | System Loop Test Facility No. 1 |
| SL-2 | System Loop Test Facility No. 2 |
| SL-3 | System Loop Test Facility No. 3 |
| SL-4 | System Loop Test Facility No. 4 |
| SMU | Structural mockup |
| SNAP | Systems for Nuclear Auxiliary Power |
| SR | Saturable reactor |
| SS | Stainless steel |
| TA | Turbine assembly |
| TAA | Turbine-alternator assembly |
| TAT | Type-approval test |
| TCL | Thermal convection loop (AGN) |
| TR | Transformer-reactor (assembly) |
| TS | Test section |
| TRW | Thompson Ramo Wooldridge |
| TSE | Test support equipment |
| VLB | Vehicle load breaker |
| VR | Voltage regulator-exciter |
| W/O | Without |
| WOO | Western Operations Office |
| -X<br>-1<br>-2<br>-3 | Standing alone (i.e., not preceded by letters of the alphabet), these designations indicate design stages of SNAP-8 hardware |

Report No. 2989

# SNAP-8 MATERIALS REPORT FOR JULY-DECEMBER 1964

by H. Derow and B. E. Farwell

Aerojet-General Corporation

## SUMMARY

Work was performed during the second half of the 1964 calendar year with the aim of providing data to guide the selection of materials for SNAP-8 system components and of providing metallurgical assistance in the design, development, fabrication, and testing of that system. The summary of this work appears below.

### Fabrication Support

A prototype -1 condenser tube-to-tube sheet joint sample representing the 0.5-in.-dia tube end was fabricated and tested. It established the feasibility of constructing a condenser by rolling and welding the 0.5-in.-dia condenser tube end followed by welding and back-brazing the 0.25-in.-dia condenser tube end. Sections of the sample were examined after welding, after stress relieving, and after exposure to a simulated 1800°F cleaning cycle and a 1950°F brazing cycle. The rolled and welded 0.5-in.-dia tube end joint was unchanged by the thermal treatments, indicating that back-brazing of the 0.25-in.-dia end would not detrimentally affect a previously rolled and welded condenser 0.5-in.-dia tube-to-tube sheet joint.

Oxidation of Stellite 6B components in air at 1600°F for one hour produced a satisfactory antigalling oxide coating for the turbine assembly labyrinth seals. The high-temperature treatment did not detrimentally affect the dimensional accuracy of the parts.

Production prototype ceramic terminal leaders, used on the speed control, were evaluated. The Koldweld (pressure weld) bond between the aluminum header housing and the copper terminal mounting flange was found to be metallurgically sound, uniform, and reproducible. Helium leak testing and pulse-echo ultrasonic inspection appear adequate for evaluating the soundness of the joint.

An argon drying train was designed, fabricated, and tested at Von Karman Center for use in furnace stress-relieving 9Cr-1Mo steel weldments. Through additional experience in local stress-relieving of several components, necessary procedural changes were made in the Aerojet-General Corporation specification covering this operation.

### System Test Support

Based on mercury capsule work at LeRC, it was decided that all mercury-loop components would be thoroughly cleaned to eliminate potential surface oil residue prior to loop assembly. Lewis Research Center work indicated that an oil film on

a boiler tube wall could result in a nonconditioned boiler which would not properly produce superheated mercury vapor. Two cleaning procedures were established utilizing an alkaline rust remover (MX-12, produced by Cee Bee Chemical Company), followed by an alkaline detergent (Alconox, produced by Alconox Inc. of New York City). One procedure involves use of an ultrasonic technique for small components; the second procedure involves use of a circulating flush technique for larger components. The PCS-1 system was cleaned using these methods.

Loop tests at AGN indicated that boiler deconditioning can be expected if a silicone oil film is deposited on the boiler mercury tube wall. In cases where such a deposit was present, it was found that alkaline cleaning would remove it but that a run-in period was required to restore the fully conditioned state. When partial surface conditioning exists, a lower outgassing pressure, approaching 5 microns, during pre-run outgassing appears to be beneficial in producing satisfactory boiler performance.

Compatibility test capsules containing combinations of thermowell materials (316 and 410 SS) and liquid indium (used as a heat-transfer medium between the thermowell tube wall and the recording thermocouple) were opened and examined after 1000 hours of exposure at 1300°F to air. No detrimental corrosion effects by the indium were noted. The indium had completely displaced itself from the pool at the bottom of the capsule and had tinned all metal surfaces. The test was restarted toward 2000 hours of total test time.

The formation, characteristics, and dissolution of solid and liquid mercury-rubidium amalgams was studied. Rubidium is a potential additive to a mercury Rankine cycle system to promote boiler conditioning for proper heat transfer, and thus proper generation, of superheated mercury vapor. Liquid mixtures of rubidium and mercury were easily produced so long as the environmental temperature was maintained above the liquidus temperature of the specific mixture composition. The chemical properties of the rubidium-mercury amalgams were found to be essentially similar to those of rubidium itself, except for a significant decrease in the reaction rates. The solid alloys are hard, brittle, metallic substances, but are completely soluble in excess mercury at room temperature so long as the resultant solution contains less than about 1 wt% of rubidium. Solid mercury-rubidium amalgams with a rubidium content up to 17.6 wt% decompose without a violent reaction in the presence of air or water at room temperature. Water produces a more rapid reaction.

At various shutdown times during component testing in rated power loop 2 (RPL-2), portions of the system piping as well as individual components were removed for analysis and evaluation of corrosion occurring during loop operation. In the NaK primary loop, a failed thermocouple well was removed and examined. It was concluded that the cross-sectional area of the weld between the end can and the thermowell tube was substantially reduced during fabrication. Failure occurred in the weld, probably due to over-insertion of the sheathed thermocouple during loop assembly. All 316 SS surfaces of the thermowell which were exposed to the NaK stream were heavily carburized.

After 800 hours of operation, a section was cut from the RPL-2 NaK primary loop at the input side of the electromagnetic (EM) pump. A surface deposit was found on the internal surface of the pipe. Analysis indicated that this deposit was primarily iron and chromium, with approximately 3-1/2 times as much iron as chromium. A layer of intergranularly deposited carbides was found on the pipe surface under the deposit. The condition of this section appeared typical of that produced by mass transfer in NaK systems operated with a high oxygen content (i.e., approximately 100 ppm or above). Such a condition existed in the RPL-2 NaK primary loop, due to a previous NaK heater failure which resulted in massive air contamination of the loop.

After approximately 45 hours of operation, a 316 SS liquid mercury filter in RPL-2 was removed and evaluated. Some deposit was found on the inside surface of the filter case and in quiescent areas inside a perforated filter element sheath. No deposit was found on the filter cloth. The particles ranged in size from 0.5 to 25 microns. The larger particles appear to be agglomerations of the smaller. Analysis of the residue showed it to consist primarily of chromium and iron, with approximately twice as much chromium as iron. Nickel, silicon, and molybdenum were also present.

The filter from the Dowtherm loop of the RPL-2 system was removed and disassembled after 45 hours of operation because there was a large increase in pressure drop across the element. A slime covered the filter element and the filter case. Analysis of this material indicated that the filter accumulation was probably due to removal by the Dowtherm of an iron oxide coating from the surface of the carbon steel loop piping. Such a coating was present when the loop was constructed.

## Lubricant-Coolant Fluid Evaluation

Analytical methods were established for evaluating the lubricant-coolant (L/C) fluid, Mix-4P3E. These methods included gas chromatographic determination of isomer ratios and volatile impurities, halogen analysis, and phenol analysis.

Compatibility test results between Mix-4P3E and copper indicate no adverse effect on either copper or Mix-4P3E in a nonirradiation, high-temperature environment.

A sample of Mix-4P3E was analyzed after a 1000-hour test of the L/C PMA. The viscosity of the fluid, measured at 75°F, decreased continually during the 1000-hour test period. The total decrease was approximately 10%. The volatile content increased slightly, approximately one-half of one percent, between the 500-hour mark and the end of the 1000-hour test period.

As a result of a meeting held with representatives of NASA, it was decided to select optimum candidate SNAP-8 lubricant-coolant fluids for comparative evaluation against Mix-4P3E. Ten different fluids were selected from a survey of the literature and a canvass of industrial sources throughout the country. All available properties of the fluids were tabulated for further analysis.

## Transformer-Reactor Assembly Heat-Sink Transition Joint

A joint was established for connecting the pure aluminum heat sink of the transformer-reactor assembly to the 304 SS piping of the lubricant-coolant loop. This joint utilizes an aluminum-to-copper Koldweld joint, plus a short stub of pure nickel TIG-welded to the copper. The other end of the nickel is TIG-welded to the 304 SS L/C loop pipe. Since the literature indicates that copper and Mix-4P3E are not compatible in a nuclear environment, it was concluded that a protective metallic coating was required over the copper to keep it from contacting the Mix-4P3E. Specimen joints were coated with three candidate metallic coatings: Electrolized chromium, electroless nickel, and silver over electroless nickel. The coated specimens were exposed to Mix-4P3E in evacuated glass capsules at 250°F. They were removed after 1000 hours of exposure for evaluation.

Evaluation was continued of an aluminum-to-copper Koldweld (pressure weld) joint to determine the effects of long-term exposure at 350°F. Specimens were examined after 1500 hours exposure. A slight diffusion zone (0.00008 in.) into the copper side of the joint interface was found. No diffusion was detected into the aluminum alloy side. Tensile tests of exposed and unexposed specimens resulted in failure of the exposed specimen at the aluminum/copper interface and in aluminum section of the unexposed specimen. The ultimate tensile strength of both specimens was approximately the same: 36,000 psi. The elongation and reduction of area measured on the aged tensile specimen was approximately 10% and 20%, respectively, of the values measured on the unexposed specimen. Thermal exposure of additional specimens was continued toward a maximum accumulated time of 3000 hours.

A coextruded tubular transition joint specimen of aluminum bonded directly to 304 SS, received from Nuclear Metals, Inc., Concord, Massachusetts, was exposed at 275°F for 350 hours. Metallographic comparison of as-received and exposed material indicates that no significant diffusion resulted from the high-temperature exposure.

## Bimetal Tube Evaluation

Flat sheet refractory bimetal tube material specimens were exposed to 1350 and 1450°F in a vacuum for 500, 1000, and 2500 hours to evaluate cross bond diffusion effects. A metallographic and hardness survey was completed. The specimens containing the direct combination of the refractory liner and the 316 SS cladding showed less diffusion effects than any of the multimetal interface systems. Tantalum, columbium, and columbium/1-zirconium refractories exhibited very thin diffusion layers at the interface with 316 SS, and showed no appreciable growth of the layer with increased temperature or time.

Material specimens were prepared for continuation of a study to develop a weld and back-brazing procedure for completing tube-to-header joints of bimetallic (refractory and 316 SS) joint elements. The tube sheet, produced by Pyromet Company using the Horton clad process, consisted of a three-layer system (columbium/iron/316 SS) with braze alloy at the two interfaces. The tube consisted of a columbium liner and 316 SS cladding.

A mercury refluxing capsule test was completed using bimetal tube capsules to evaluate tube butt weld joining procedures. The welds were contained both in the lower portion of the capsule (exposed to liquid mercury) and in the upper portion of the capsule (exposed to condensing mercury vapor). Both welds of a "control" 9Cr-1Mo steel unclad capsule showed no evidence of mercury corrosion. In two capsules fabricated from 9Cr-1Mo steel/316 SS bimetallic tube which were run, the welds exposed to liquid mercury showed no mercury corrosion. Corrosion occurred in the weld exposed to the condensing mercury vapor in one of these capsules. Through-wall penetration by the 316 SS weld metal had occurred at this location during fabrication. The welds in a columbium/316 SS bimetal tube were produced by controlled partial penetration using an electron beam welding source. The intentional non-penetration into the columbium liner produced a gap at the unjoined columbium liner interface. In spite of this gap, corrosion of the exposed 316 SS weld had not occurred.

## 9Cr-1Mo Steel Strength Evaluation

Creep rupture tests were conducted on welded and unwelded 9Cr-1Mo steel specimens to evaluate the gross creep rupture strength efficiency of 9Cr-1Mo steel welds. The tests were run at 1325°F at two stress levels - 1600 and 2300 psi. The test results indicate that welding does not decrease the creep or creep rupture strength of 9Cr-1Mo steel at 1325°F.

Weld transition joints between 9Cr-1Mo steel and 316 SS, using 310 SS weld wire, were tested at 1300°F to grossly evaluate the strength of the joint. The test exposure consisted of a simulated system startup test (25 short-period temperature cycles between ambient and 1300°F) and a simulated system service life test (continuous exposure at 1300°F with a stress cycle every 8 sec). Two specimens were tested: one in the as-welded condition and the other containing a weld which had been roll planished to refine and increase the strength of the weld structure. Both specimens successfully completed the simulated system startup tests. The simulated system life test was terminated after 2227 hours because of excessive bulging in the 9Cr-1Mo steel section of the weld planished specimen. The as-welded specimen gave no indication of yielding. It is concluded that there is no hazard of failure by creep in a weldment between 9Cr-1Mo steel and 316 SS using 310 filler metal. The bulging of the 9Cr-1Mo steel section in the weld planished specimen appeared to have resulted from overstressing of the material caused by excessive wall thinning produced during the planishing operation.

A trial run was completed to evaluate both the equipment and a testing procedure for producing decarburization and grain growth in the surface of 9Cr-1Mo steel exposed to NaK in a cyclic creep test. The procedure and equipment proved adequate. Final preparations were made for initiation of the test on 9Cr-1Mo steel specimens to grossly evaluate the effect of cyclic creep conditions in a NaK environment on the creep strength of the material.

## Corrosion Loop Program

The assembly and installation of CL-4 was completed during the report period. The design and operating parameters of a hydrogen injector system for use in CL-4 were completed, and the ordering of materials and components for system fabrication was started. The fabrication of the liquid corrosion product separator for CL-4 was completed. This separator, fabricated from 9Cr-1Mo steel, utilizes columbium and iron wool as the separator medium. Checkout of the instrumentation and electrical hookup was started.

After a total mercury boiling time for CL-3 of 4400 hours, test operations were discontinued and preparations were made for evaluation of the loop materials and components. The mercury boiler in this loop required a run-in period to attain rated performance. The primary and condensing NaK systems of CL-3 operated satisfactorily until the time of final loop shutdown on 23 December. At this point, a leak developed in the vicinity of the NaK heater. The operating history with respect to oxide control of the primary loop indicates that the oxide level was maintained at 38 ppm or less during operation. Cold trapping was performed several times using the NaK purification system, to reduce the oxide level from the peak of 38 ppm to 18 ppm. Several loop shutdowns occurred during the operating history of CL-3 for various reasons, including maintenance, power failures, and auxiliary equipment failures such as instrumentation leaks. All restarts produced immediate superheating of the mercury vapor.

Component test loop 2 (CTL-2) was operated to conduct mercury vapor phase corrosion product separator tests. An impingement-type separator was tested. During the test run, the boiler outlet pressure (the criteria for filter evaluation) increased from 250 psig to 300 psig in 10 hours due to corrosion product buildup on the adjustable choke nozzle downstream of the filter. During the preliminary base run, the same increase in boiler outlet pressure occurred in 16.7 hours, indicating that the impingement separator was not effective.

## I. INTRODUCTION

The objectives of the SNAP-8 Materials Program are to provide data to serve as a basis for the selection of materials for use in various components; to assist, through metallurgical studies, in the design, development, fabrication, and testing of the SNAP-8 system; and to evaluate the resistance of the reference material (i.e., 9Cr-1Mo alloy steel) to mercury corrosion. This report covers the work performed during the second 6 months of the 1964 calendar year.

## II. FABRICATION SUPPORT

### A. BOILER

Boilers A-3 and A-4 were degreased prior to stress relieving to remove any internal oil or particulate surface contamination. A procedure slightly modified from that used for boiler A-2 (Reference 1) was used. The procedure modifications were as follows:

1. With the boiler in the vertical position and the mercury inlet at the top, the tubes were individually power flushed by condensed, hot trichloroethylene (TCE).

2. The tubes were then filled with TCE and gravity flushed.

3. The tubes were dried by room temperature nitrogen purge.

4. The boiler was inverted so that the mercury outlet end was on the top, and steps 1 through 3 were repeated.

5. The boiler was inverted once more to the original position and only steps 1 and 3 were repeated. Each tube was capped to prevent inadvertent recontamination.

### B. CONDENSER

During the report period, difficulty was experienced in development of a satisfactory rolling procedure for the 0.25-in.-dia end of the condenser tube-to-tube sheet joint. A potential fabrication procedure which eliminates this operation is to roll and weld the 0.5-in.-dia tube end (a satisfactory rolling technique has been established for this joint) and weld and back-braze the 0.25-in.-dia tube end. To determine the effect of the brazing operation on the 0.5-in.-dia tube rolled and welded joint, a seven-tube qualification sample was fabricated and evaluated. Sections were taken from the sample after welding, after stress-relieving, and after exposure to a simulated 1800°F pre-braze cleaning cycle and 1950°F brazing cycle in a hydrogen atmosphere. The joint configuration was unchanged by the thermal treatments, indicating that the proposed alternate procedure is feasible. Additional development work is required, however, to optimize the backbrazing operation since the results of backbrazing full-sized SNAP-8 condensers has not been entirely satisfactory.

## C. TURBINE ASSEMBLY

A surface oxide coating on TA seals was selected to increase the resistance to galling of Stellite 6B seals in the event they should contact the turbine rotor hubs during TA operation. Previous experience by the Stellite Division of Union Carbide and the estimated difference in surface energy of a metal and its oxide indicated that a surface with an opaque tightly adherent oxide coating rubbing against a nonoxide surface was desirable for minimum galling.

Samples of the alloy were processed to establish a procedure for producing the desired oxide coating. The samples were heated in an air atmosphere, held at various temperatures for 1 hour, and then air cooled. The following results were observed:

Surface Oxidation of Stellite 6B After 1 Hr at Temperature in Air

| Exposure Temperature (°F) | Metal Surface Color at Room Temperature After Exposure |
|---|---|
| 1200 | Translucent blue |
| 1400 | Translucent green-blue |
| 1500 | Semi-translucent brown-green |
| 1600 | Opaque black-brown |

All of the oxide coatings were tenacious; however, a minimum temperature of 1600°F was required to produce the desired opaque coating.

A TA labyrinth seal was oxidized at 1600°F and cooled to 700°F in the furnace (with furnace power off). Inspection before and after the exposure indicated there was no adverse effect on dimensional tolerances.

## D. ELECTRICAL COMPONENTS

### 1. Speed Control

Seven production prototype ceramic terminal leaders to be used on the SNAP-8 speed control were evaluated to determine the quality of the Koldweld bond between electrolytic tough pitch copper and 6061 aluminum alloy. The Koldweld process is a proprietary pressure-welding procedure of Kelsey-Hayes. It was concluded that the bond was metallurgically sound, uniform, and reproducible. It was also concluded that the bond can be inspected and evaluated by helium leak testing and by employing pulse-echo ultrasound. A technical memorandum (Reference 2) was published to document the findings.

A mockup section of the -1 speed control module was prepared to evaluate weld assembly sequence and weld heat sink tooling. The lower level section of the assembly was successfully welded. Proper temperature control was maintained during welding. The partially completed unit was photographed as an assembly aid (Figure 1).

### 2. Transformer-Reactor Assembly (TRA)

A welding procedure was developed and samples were prepared of joints between copper braid and copper bar. These joints are used in the TRA harness design. These samples were sectioned and it was determined that the procedure used provided satisfactory weld penetration as shown in Figures 2 and 3.

A welding procedure was developed for joining of rectifier terminals (diodes) of the TRA. The two terminal materials joined are alloy 52 (52% Ni, 48% Fe) and oxygen-free copper. Metallographic samples (Figure 4) indicated satisfactory joints. They are primarily TIG-brazed; however, a small amount of fusion occurs. To facilitate assembly of the diodes, a dummy section of the -1 speed control module was prepared. Using this dummy, an assembly sequence and weld fixture for the module was developed. Thermocouples were attached to several diodes to determine the temperatures reached at sensitive portions of the diodes during welding. The heat sinks of the weld fixture satisfactorily maintained diode temperatures below 300°F (the permissible maximum).

## E. BELLOWS

Bellows assemblies are being fabricated by the Palmer Company. They will operate at 700°F with an internal pressure of 20 psi. This component contains weldments of AISI 4340 steel and 9Cr-1Mo steel. A final closure weld of 9Cr-1Mo to itself is required. The standard SNAP-8 stress relieving temperature for 9Cr-1Mo weldments is 1350 ±25°F; however, the $A_{C1}$ temperature for AISI 4340 may be 1300°F or slightly lower, depending on chemistry variations and fabrication history. If the temperature of AISI 4340 components during stress relief exceeds the $A_{C1}$ temperature, martensite transformation occurs during cooling. The impact strength and ductility of the 4340 would be reduced and could lead to service failures. Although an extra stress relief at a lower temperature will temper the martensite and restore satisfactory material properties, it appeared that the stress relief of the final weldment on the bellows should be performed at 1225 ±25°F - not 1350 ±25°F. The use of this temperature avoids possible martensite formation in the AISI 4340 and the necessity of a double stress relief.

To determine the effect of the 1225°F stress-relieving temperature on the 9Cr-1Mo and on the AISI 4340, a series of stress-relieving treatments was performed on welded 9Cr-1Mo tube (1 in. OD by 0.109 in. wall) and welded AISI 4340 plate, 3/4-in. thick. Table 1 presents the stress-relieving conditions and the results of the tests. On the heat of AISI 4340 investigated, weld metal softening continued through 1350°F and hardening occurred at 1400°F. The one-point increase in hardness as measured on the 1275°F specimens, is considered to be within experimental error and not indicative of hardness. Definite hardening of the AISI 4340 parent metal occurred when the 1400°F stress-relieving temperature was employed.

It was concluded that a 1225°F stress relief of the 9Cr-1Mo steel would not be detrimental. There is a hardness increase over that resulting from the standard 1350°F stress relief. But it is not judged to be detrimental in view of the satisfactory ductility apparent from the flattening test.

## F. STRESS-RELIEVING OF 9Cr-1Mo STEEL COMPONENTS

### 1. Furnace Stress Relieving

An argon drying train was designed and fabricated to provide a satisfactorily pure argon atmosphere for stress-relieving SNAP-8 components fabricated at VKC from 9Cr-1Mo steel. The unit consists of a molecular sieve dryer followed by a hot (1000°F) 50-50 titanium-zirconium alloy chip getter. Manifold argon with a dewpoint of -55°F is passed through the dryer and the hot getter at a rate of 60 cu ft/hr and into a 54 cu ft capacity weld sealed retort. After purging of the retort, the dewpoint of the exiting argon is -86°F. 9Cr-1Mo samples were suspended in the retort, heated to 1350°F, held for 1 hour at temperature, and cooled in the argon atmosphere to test the adequacy of the system. They were visually unchanged in surface appearance after the treatment.

### 2. Local Stress Relieving of Weld Joints

The 9Cr-1Mo Conoseal flange welded joints of the A-3 boiler and A-1 and A-2 mercury PMA were locally stress-relieved in accordance with Reference 3 using induction heating. To avoid inductor overheating, which occurred occasionally in the past, valve and line restrictions in the incoming water coolant lines were removed.

Induction heating was also employed to stress-relieve welded joints of 9Cr-1Mo to 316 nipples on two Chempump mercury inlet and outlet lines. The pump bodies were maintained below 150°F during the 1350°F stress-relieving operation by flowing water through copper tubing wrapped around the base of the inlet and outlet mercury lines.

The experience of performing the stress-relieving procedure described in Reference 3 on these additional components led to revision of the specification. The thermocouples are attached on the outer diameter of the weld joint by capacitance discharge welding rather than to the inner diameter of the weld joint for greater accuracy in temperature measurement. Provision for additional cooling of Conoseal flanges through use of an internal cooling coil was added for use in the event that for some configurations the 500°F maximum allowable temperature could not be maintained.

An electrical resistance clamshell heater was employed to locally stress-relieve a welded 9Cr-1Mo to 316 joint at 1350°F for 1 hour on the mercury inlet line of RPL-2. Argon was flowed through the boiler inlet line during heating and cooling.

## G. SPECIFICATIONS

The following specifications were issued or modified during the report period:

AGC-10227B - "Welding, Fusion, SNAP-8 Materials"
AGC-10227/1 - "Welding, Fusion, (9Cr-1Mo) Stainless Steel"
AGC-10227/2 - "Welding, Fusion, 316 Stainless Steel"
AGC-10227/3 - "Welding, Fusion, 410 Stainless Steel"
AGC-10227/4 - "Welding, Fusion, (9Cr-1Mo) Stainless Steel to 316 Stainless Steel"
AGC-10227/5 - "Welding, Fusion, Haynes Stellite 6B"
AGC-10228A - "Tube to Tube Sheet Welds, Inspection of"
AGC-10319 (Amendment No. 1) - "Cleaning SNAP-8 Components and Systems, Procedure for"
AGC-10319/2 - "Degreasing of Non-Precision SNAP-8 Components and Systems, Procedure for"
AGC-10319/5 - "Cleaning of SNAP-8 Thrust Bearing Assembly Components (P/N 122 C16)"
AGC-10331 - "Electrical Connections, Inert-Arc Welding of, Procedure for"
AGC-10354 - "Development Material Specification, Steel, Bearing, AISI M50, Requirements for"

# III. SYSTEM TEST SUPPORT

## A. BOILER CONDITIONING STUDIES

The objective of this task is twofold: (1) to evaluate the effect of various tube surface conditions on the capacity of the SNAP-8 boiler to produce superheated mercury vapor on system startup, and (2) to establish procedures for removing any surface residue found to be detrimental to boiler conditioning.

### 1. Preassembly Cleaning of Loop Components

Mercury capsule experiments at LeRC indicate that oil films as thin as 6 angstroms (approximately $2 \times 10^{-8}$ in.) may prevent proper heat transfer into the flowing mercury in the SNAP-8 boiler. As a consequence, all SNAP-8 PCS-1 components were cleaned to remove any oil residue. The established cleaning procedure utilizes two alkaline cleaning solutions. The first is MX-12, an alkaline rust remover manufactured by Cee Bee Chemical Co., Downey, Calif., which contains approximately 70% sodium hydroxide. This solution acts both as a rust remover and as an excellent oil remover. The sequestering and wetting agents in the solution give it low surface tension and facilitate intimate contact of all the metal surfaces. The second solution is Alconox, an alkaline detergent produced by Alconox, Inc., New York, N.Y. This detergent has a very low surface tension, and a great affinity for oil residue. The procedures utilized are described in Appendix B of Reference 4. One is an ultrasonic technique for smaller components (such as valves and bellows) which can fit into available ultrasonic equipment.

A second procedure utilizes a circulating flush and is applicable to lines, boiler, condenser, and turbine simulator. The PCS turbine and mercury PMA are not cleaned in this fashion, but cleanliness is maintained through ultrasonic cleaning in Freon of individual parts during assembly.

### 2. Capsule Tests

Several chemical solutions were evaluated as agents for removing varnish residue resulting from heating SF-97 silicone oil in an evacuated and sealed glass capsule (the contaminating oil of RPL-2) for 5 hours at 1150°F. After approximately 1 hour of immersion at near its boiling point, an MX-12 solution containing sequestering and wetting agents removed the residue. (The solution used was equivalent to Type I solution in accordance with MIL-C-14460, but related solutions such as Cee Bee Chemical Company's MX-12 alkaline rust remover is adequate.) A solution of Oakite 90 alkaline solution (8 oz/gal) removed approximately half of the residue after 2 to 2-1/2 hours of exposure (at near the boiling point). Other solutions that were tried but were unsuccessful were Turco's "Jetisoil-NP," OLIN alcoholic potassium hydroxide, and Freon-PCA. Although the Freon did not appear to remove the baked-on residue, it did appear that adhesion of the residue on the capsule wall was reduced.

Freon-PCA was evaluated as an agent for removal of unreacted silicone oil SF-97 from 9Cr-1Mo steel surfaces. A polished metal specimen was coated with the silicone oil. The surface was then scanned by infrared spectrophotometer and the results automatically plotted. Approximately 10 to 15 ml of Freon PCA was then put onto the specimen dropwise from an eyedropper. A scan of the surface after the wash revealed no silicone oil IR pattern. This method can identify a minimum film thickness in the range from 1 to 10 microns (approximately 4 to 40 x $10^{-5}$ in.), much thicker than that deemed detrimental as a result of capsule tests at LeRC.

### 3. Loop Tests

A series of test runs was made with CTL-2 (fabricated from Haynes 25) at AGN to determine if silicone oil could affect the performance of a mercury boiler and if the oil could be removed adequately with chemical cleaners. This program was conducted after the mercury vapor corrosion product separator tests described in Section VIII,C. The CTL-2 boiler consists of a static tank of NaK with the mercury coil and the five 10-kw electrical Calrod-type heaters immersed in the NaK as shown in Figure 5. Since the NaK control thermocouple is located in the middle of the NaK tank and mixing of the NaK depends on natural circulation, it was estimated that the NaK temperature at the top of the tank (outlet of the mercury boiler coil) was 50°F higher than indicated by the control thermocouple.

A detailed description of the tests appears in Section VIII,J,2 of Reference 4. A summary of the results is given in Table 2. The boiler performance was judged by the boiler outlet temperature and pressure, the pressure drop across the boiler coil, and the power input to the boiler. As noted in Table 2, the pressure drop across the boiler was 30 psi or greater when the boiler was conditioned. The heat required to boil 1050 lb/hr of mercury at a saturation temperature corresponding to 270 psia is approximately 45 kw. In the runs when the boiler was deconditioned (B, C, D, and G in Table 2), the heat that could be put into the boiler without exceeding the NaK temperature limit (1360°F) was less than half the amount required to achieve 100% quality.

The following conclusions were drawn from this series of test runs performed with CTL-2:

a. The addition of small amounts of silicone oil to the boiler using Freon as a solvent causes the CTL-2 boiler to degrade in performance or to become deconditioned.

b. Cleaning the CTL-2 boiler with alkaline cleaners, MX-12 and Alconox, did not immediately restore the boiler performance. Some operation time was necessary before the boiler performance was restored and the boiler was conditioned.

c. Decrease of the pressure in the boiler coil from 500 microns to 5 microns during the pre-run outgassing period did not affect boiler performance if the boiler was fully conditioned or deconditioned. However, if the boiler was partly conditioned, a lower outgassing pressure appeared to be beneficial to boiler performance.

## B. INDIUM/THERMOWELL COMPATIBILITY

A pool of indium is being employed in thermocouple wells in SNAP-8 loops as a heat-transfer medium between the thermocouple well and the thermocouple sheath. An indium/thermowell material compatibility test was initiated to determine the effect upon thermocouple well and sheath materials of exposure to indium at 1300°F. Wires of 316 SS and 410 SS, representing the thermocouple sheath, were placed in thermowells of 316 SS and 410 SS. All possible thermowell-wire-material combinations were tested. The well was filled with indium to a depth of 1/2 in. A cap was then welded on the open end of the thermowell to provide a sealed test capsule. The atmosphere inside the capsule was air. Capsules were removed from test after 1000 hours of exposure. A metallographic examination was made. No reduction in wire diameter could be detected, and no evidence of corrosion was found. No metallurgical changes were noted excepting the expected carbide precipitation in 316 SS and spheroidization in 410 SS. The indium had wet all interior surfaces of the capsule. Consequently, there was no longer a pool of indium present, but rather a surface film. The capsules were resealed and the test resumed toward 2000 hours of exposure.

The preliminary analysis indicates that the thermowell and thermocouple sheath materials are compatible with indium but that some difficulty may be experienced in maintaining an indium pool in the thermowell.

## C. MERCURY-RUBIDIUM AMALGAMATION

Rubidium is a potential additive to a mercury loop operated as a Rankine cycle system to promote mercury boiler conditioning (for proper heat transfer). The selection of rubidium was based on evaluation of its physical properties and pool-boiling experiments conducted at AGN (Reference 5). Various mercury-rubidium compositions of rubidium contents up to about 21 wt% (see Reference 6) were evaluated for formation and dissolution characteristics of solid and liquid amalgams. Potential cleaning techniques were evaluated for removal of solid amalgam plugs, were they to form in a loop. The program is described in Section VIII,D in Reference 4. The general observations made during the study and the conclusions reached are discussed below.

Liquid amalgams of rubidium with mercury, containing 1 wt% or less Rb, are easily formed and homogenized at an ambient temperature of 110°F using simple agitation. Those amalgams whose liquidus temperature is above this ambient require heating to the liquidus and some agitation before an homogeneous liquid amalgam forms.

The chemical properties of the rubidium-mercury amalgams studied were similar to those of pure rubidium except for a notable decrease in reaction rates. Thus, the amalgams may be handled in air and even decomposed in water without undue hazard or violence, as contrasted with the virtually explosive character of pure rubidium in similar environments. As solids, the amalgams containing a minimum of 4.1% Rb ($RbHg_{10}$) are hard and brittle. All the solid amalgams were completely soluble in excess mercury at an ambient temperature of 77°F to form solutions of less than about 1% Rb. A rubidium oxide or hydroxide coating on the surface of the solid was not soluble in mercury, and agitation was necessary to break through the protective coatings to effect dissolution of the Rb-Hg amalgam.

Mercury-rubidium amalgams of up to about 21 wt% rubidium can be safely decomposed in the presence of air with water at room temperature without ignition of the hydrogen formed: the decomposition products of rubidium are soluble in the water, leaving the clean mercury behind. The amalgams can also be handled safely in air with no sparks or fire; however, they slowly deteriorate due to a rubidium reaction whenever an amalgam is exposed to air and water vapor.

Water or humid air can be safely (assuming that normal precautions are taken to adequately vent evolved hydrogen gas) and freely utilized to decompose rubidium amalgam deposits or plugs in SNAP-8 hardware except in cases where the released mercury cannot drain away from the amalgam (to allow fresh exposure of rubidium). Mercury can be used to dissolve plugs; however, some method of breaking down large plugs would be necessary to minimize the required time - perhaps abrasive agitation. Heating to approximately 500°F will melt the plug.

## D. RPL-2 EVALUATION

SNAP-8 components were tested in the Rated Power Loop No. 2 (RPL-2) at system design conditions. The RPL-2 included a primary NaK loop, a mercury loop, a heat-rejection loop, and a lubricant-coolant loop. This test fixture was used to provide basic operating data for component and materials evaluation.

### 1. NaK Primary Loop

A failure in a thermocouple well at the NaK heater outlet of RPL-2 was investigated. Failure occurred in the weld between the end cap and the thermowell tube. The original cross-sectional area of the weld had been substantially reduced during fabrication. Weld failure probably occurred because of excess force applied through over-insertion of the sheathed thermocouple during loop assembly. It was also found that all 316 SS surfaces of the thermowell which were exposed to NaK were heavily carburized to a depth of 0.001 in. A technical memorandum (Reference 7) was published to document the results of this investigation.

During the RPL-2 shutdown for conversion to TAA testing, after approximately 800 hours of operation, a section was cut from the NaK loop at the input side of the EM pump. The pipe section included the circumferential butt weld used for installation of the pump into the loop. The interior of the removed section was coated with a loosely adherent layer of particles which visually appeared to be crystalline. Metallographic sections of the pipe are shown as Figures 6, 7, and 8. Figure 6 is typical of the appearance of all straight sections of pipe which were examined.

The deposit layer was approximately 0.006 in. thick. It appeared as an agglomeration of particles with a great many interspersed voids. The internal surface of the tube, under the deposited layer, contained a layer of intergranularly deposited carbides approximately 0.001 in. deep.

The penetration of a circumferential weld contained in the specimen exceeded 100%, and the dropthrough produced a rough jagged interior surface with protuberances approximately 0.008 to 0.010 in. high. The surface deposit was heavier in the weld area, as can be seen by comparing Figures 6 and 8. Note that the buildup of deposit is heavier on one side of the protuberances than on the other. The orientation of the removed pipe section was not identified, and thus it is not known whether the increased buildup occurred at points of impingement (on the upstream side of the weld) or at points where the fluid was more quiescent (on the downstream side).

Samples attained by scraping the deposit from the pipe wall were examined by X-ray diffraction. The only diffraction pattern lines which were detected were characteristic of $\alpha$ iron, chromium, or nickel. Semiquantitative emission spectroscopy was performed. The principal constituents were iron and chromium, with approximately 3-1/2 times as much iron as chromium.

The condition of this section is typical of that produced by mass transfer in NaK systems operated with a high oxygen content (i.e., approximately 100 ppm or above). The ORNL found similar mass transfer of metallic elements and carbon in high-oxygen containing loops run under a NASA-sponsored SNAP-8 support program, wherein the loop design was a scaled-down version of the SNAP-8 primary loop.

### 2. Mercury Loop

The boiler tube surface (9Cr-1Mo alloy steel) was oxidized during final stress-relieving because of an air-contaminated argon furnace atmosphere.

It was decided to use the boiler with the oxide layer for two reasons. First, the alternative of removing the oxide by chemicals presented cleaning problems. Second, the TS-2B boiler (9Cr-1Mo steel) had produced superheated mercury vapor after exposure of internal mercury tubes to flowing air for

1 hour at 1300°F. This exposure would have resulted in an oxide layer similar to that present on the boiler under consideration. A vacuum stress-relieving operation was called for on subsequent boilers to be fabricated to avoid the oxidized tube surface.

A large increase in pressure drop occurred across the liquid mercury filter of RPL-2 after approximately 45 hours of operation. The case and the element, fabricated of 316 SS, were removed from the loop and evaluated.

All surfaces which had been exposed to mercury were darkened. Analysis is in process to determine the nature of this darkened layer. Some deposit was found on the filter case in the form of a ring at what had apparently been the liquid level. The construction of the filter is such that a partial liquid inventory would tend to be established in the presence of non-condensable gases. This condition is aggravated at a flow rate lower than design. This filter did experience reduced flow. Deposits were also found in quiescent areas inside the perforated element filter sheath above the top of the filter cloth. The location of deposits is shown in Figure 9. No material deposit was found on the filter cloth. The absence of material deposit on the cloth is attributed to a backflush of the filter which automatically occurs when the mercury is drained from the loop.

The particles, which were recovered by scraping, were from 0.5 to 25 microns. Most of the smaller particles (i.e., 0.5 to 3 microns) were roughly globular. The larger particles appeared to be agglomerations of the smaller particles.

The total amount of material recovered was insufficient for gravimetric analysis. An X-ray diffraction powder pattern indicated the presence of $Fe_2O_3$ and/or CrO. Other peaks were present but were too weak to interpret. Emission spectroscopy showed the residue to consist primarily of chromium and iron with approximately twice as much chromium as iron. Nickel, silicon, and molybdenum were also present in approximately equal parts - each about one-half the amount of iron.

The principal constituents of 9Cr-1Mo steel are iron and chromium. Chromium is more soluble in mercury than iron. It is therefore concluded that the composition of the deposit is indicative of corrosion of 9Cr-1Mo steel components in the system, the boiler, and/or the condenser. These corrosion products are apparently carried around the loop and ultimately lodge in the liquid Hg filter. Evaluation of these components at the conclusion of RPL-2 operation is required to ascertain the extent of corrosion and to extrapolate the amount of corrosion anticipated in 10,000 hours (the required life of the SNAP-8 system).

### 3. Dowtherm Loop

The RPL-2 Hg loop utilizes an Hg chempump, manufactured by Chempump Division of Fostoria Corp., in lieu of a SNAP-8 Hg PMA which is still under development. The chempump is cooled by a separate coolant loop utilizing Dowtherm E.

A large increase in pressure drop occurred at the Dowtherm loop filter after approximately 45 hours of operation. The case was removed and a slime was recovered from the filter element and from the bottom of the filter case. The specimen was moist with Dowtherm. Analysis of this material was performed with the following results:

a. Infrared spectroscopy indicated the presence of carbonates.

b. Microanalysis indicated the residue contained 18.4% carbon, 1.4% hydrogen, and a combustion residue of 68.4%. The source of the carbon and hydrogen is assumed to be from Dowtherm. The large percentage of combustion residue (inorganic ash) indicates that the material is the product of contamination or corrosion and not from polymerization or breakdown of the fluid.

c. Semiquantitative analysis of the combustion residue by emission spectroscopy indicated that the major elements present were iron and chromium. Nickel and silicon were also detected, but to a lesser degree.

d. Qualitative analysis gave no indication of chloride. Corrosion by the Dowtherm mixture should produce chlorides since Dowtherm E is about 5 wt% chloride and will hydrolyze if contaminated with water.

It was concluded that the filter clogging was not due either to corrosion or to fluid polymerization, but rather to the action of Dowtherm in removing contaminants from the loop walls. The loop piping was carbon steel and contained a tightly adherent black iron oxide coating as received from the mill. The pipe was degreased, but no attempt was made to remove the oxide coating prior to loop fabrication.

## IV. LUBRICANT-COOLANT FLUID EVALUATION

### A. FLUID ANALYSIS PROCEDURES

Analytical methods for evaluating the Mix-4P3E were established through a laboratory program which was completed during the report period.

#### 1. Gas Chromatographic Determination of Isomer Ratios and Volatile Impurities

A method was selected for identification and quantitative analysis of the isomer distribution in the fluid, and to establish the identity and quantitative distribution of volatiles and possible homologues.

Three potential partitioning fluids were evaluated. These were Dow Corning DC-710, Dow Corning High Vacuum Grease, and General Electric SE-52. The DC-710 fluid was unsatisfactory because it resulted in "bleeding," which is the emission of volatiles from the column fluid caused by thermal breakdown. These volatiles fog the results of the volatiles contained in the Mix-4P3E. The SE-52 fluid was selected because it gave better and sharper resolution of the isomers than the High Vacuum Grease. The developed procedure will be issued as part of Aerojet Standard AGC-STD-2003, which covers sampling and testing of chemicals and materials.

### 2. Halogen Analysis

An analysis procedure was established for determination of total halogen content. The method is a modification of that contained in Reference 9. A toluene solution of the sample is refluxed with sodium metal in the presence of a small amount of butanol. This converts the halogens to an easily ionized sodium salt. Reproducible results were obtained during the test program. This method will be incorporated into the Aerojet Standard AGC-STD-3003 (as Method 5604), which covers determination of halogens in organic compounds.

### 3. Phenol Analysis

Phenols and other acidic materials in the Mix-4P3E will be determined by nonaqueous titration. This method consists of dissolving the sample of Mix-4P3E in pyridine and titrating to a visual or potentiometric endpoint with a standard solution of potassium hydroxide in isopropyl alcohol (nonaqueous solution). The procedure is being incorporated into AGC-STD-3003 (as Method 5106.1), which covers determination of very weak acids in organic compounds by nonaqueous titration.

## B. MATERIALS COMPATIBILITY

Qualitative results of tests wherein copper was exposed to two samples of Mix-4P3E were reported in Section VI, Page VI-23, of Reference 1. All fluid was from lot 12-2P supplied by Dow Chemical Co. One fluid sample was used as-received, and contained approximately 2% volatile impurities. The other sample was acquired by distilling off these volatiles. Final tests on the fluid were completed during this report period. The following is a synopsis of the results:

1. The weight change of the copper specimens was negligible and there appeared to be no pitting or roughening. Some of the specimens had discolored with definite demarkation lines at the Mix-4P3E liquid-vapor interface. The specimens that had greatest color change were those exposed to the as-received Mix-4P3E with the higher volatile content.

2. Both samples had fluid darkened during exposure, but no apparent degradation was detected by gas chromatographic analysis.

3. The acidity of the two fluid samples did not change significantly.

It is concluded as a result of these tests that in a nonirradiation, elevated temperature exposure, Mix-4P3E does not adversely attack copper, and copper does not cause degradation of Mix-4P3E.

## C. POST-TEST FLUID ANALYSIS

Subsequent to a 1000-hour test of the L/C PMA at TRW, a sample of the Mix-4P3E (Dow's ET-378, Lot 43-2) was returned to VKC to determine if the properties had been affected. The density, pour point, infrared data, and the isomer distribution were the same as the original and 500-hour specimens which had been evaluated previously. The viscosity exhibited a continued decrease during the entire 1000-hour test period. The volatile content, which showed no change during the first 500 hours, increased slightly during the second 500 hours. The analysis indicated that these volatiles were very light. The changes which occurred during the 1000-hour test period are shown in Table 3. They are not excessive and it is judged that the 1000 hours of operating time had no adverse effect on the fluid.

### D. GENERAL CONSIDERATIONS

A periodic summarization covering investigative studies with Mix-4P3E (the SNAP-8 lubricant/coolant fluid) was issued as a technical memorandum (Reference 10). The memorandum covered the period from 1 January through 15 July 1964. The goal of this investigation was (1) to supplement available manufacturer's data where necessary, and (2) to evaluate the effects on the fluid of system component tests. Information that was promulgated appears below.

A procurement specification (AGC-10320) was issued

Analytical methods and purification techniques were established

Selected environmental tests were run. Analysis was conducted on samples taken during testing of various SNAP-8 components.

The results of SNAP-8 experience indicate that some of the characteristics of this fluid may prove undesirable for SNAP-8 use. For example, the fluid dissolves relatively large amounts of air or other gases leading to a tendency of the fluid to foam under test conditions. Also, the supercooled fluid crystallizes under undefined conditions if the isomer ratio is not within a specific range. Additionally, the lubricity of the fluid appears marginal.

A meeting was held with representatives of NASA to discuss the use of this fluid. It was deemed advisable to select several alternate candidate fluids as backup to Mix-4P3E if further experience showed it to be unacceptable. An action item resulting from this meeting was that the Materials Section was directed to compile a list of other potential SNAP-8 lubricant/coolant candidate fluids. These would then be analyzed in detail to select optimum candidates for comparative evaluation against 4P3E.

Ten different fluids resulted from a survey of the literature and of experts in universities, industrial organizations, and Government laboratories. These fluids are described in Table 4. All available properties of these fluids were tabulated from the various available sources.

## V. TRANSFORMER-REACTOR ASSEMBLY HEAT SINK TRANSITION JOINT

The objective of this task is to establish an adequate joint for connecting the heat sink of the transformer-reactor assembly to the line of the lubricant/coolant loop. The heat sink is fabricated of pure aluminum and the piping of the lubricant/coolant loop is 304 SS.

## A. JOINT CONCEPT

Direct joining of aluminum to stainless steel by welding is not an established state-of-the-art procedure. Several companies are working on the problem. One company - Nuclear Metals, Inc., Concord, Mass. - provided samples of a hot coextruded transition joint to Aerojet-General for evaluation. An acceptable transition joint using state-of-the-art procedures was made by joining aluminum to copper using the Koldweld (pressure welding) process of Kelsey Hayes, then joining copper to nickel by TIG-welding, followed by joining nickel to 304 SS by TIG-welding. Previously prepared lap and butt weld samples had indicated (Section VIII,A of Reference 11) that the copper end of the coldwelded part should not be welded directly to the 304 SS. It was concluded that a nickel transition piece between the Cu and 304 SS is necessary.

There is evidence that copper is attacked by the coolant fluid (Mix-4P3E polyphenyl ether) if the system operates under nuclear radiation. Because of the potential copper corrosion problem, it is necessary to apply a protective metallic coating over the copper to keep it from contacting the Mix-4P3E.

## B. PROTECTION OF COPPER IN Al/Cu TRANSITION JOINT

A testing program (see Section VIII,C in Reference 4) was initiated to evaluate three optimum candidate coatings by exposing coated specimens to Mix-4P3E heated to 250°F in evacuated ($10^{-3}$ to $10^{-4}$ torr) glass capsules. Tubular transition joint specimens of Al/Cu/Ni/304 SS were prepared for coating evaluation. The Cu/Ni joint was completed using Monel (70Ni/30Cu) filler metal and the Ni/304 SS joint was completed using Inco A filler metal. Figure 10 shows a sketch of the joint specimen.

The coatings and the plating vendors who applied them to the specimens are shown below.

Copper-Protecting Coatings for T/R to L/C Loop Transition Joint

| Type | Thickness | Specification | Vendor |
|---|---|---|---|
| "Electrolized" chromium | Approximately 0.4 mil | QQ-S-320, Cl. 2 | Electrolizing Sales Inc., Los Angeles |
| Electroless nickel | 1 to 2 mils | MIL-C-26074, Cl. 1 | Hixson Metal Finishing, Newport Beach, Calif. |
| Silver over electroless nickel | Approximately 0.5 mil silver over 1 to 2 mils of electroless nickel | Silver per QQ-S-365 Type 1, Class 1; electroless nickel per MIL-C-26074, Cl. 1 | Hixson Metal Finishing, Newport Beach, Calif. |

Each as-received plated specimen was visually examined to ascertain the condition of the deposited coating. A synopsis of this examination appears below.

### 1. "Electrolized" Chromium

Coatings were smooth and lustrous. Small depressions associated with welds did not appear to be coated. Some of the areas inside the tubes did not appear to have a coating, especially on the aluminum. The vendor had stated the poor internal coating was caused by an inadequately deoxidized surface. A proper surface was difficult to achieve because the tube inside diameter was too small.

### 2. Electroless Nickel

The coating was uniform, lustrous gray-silver, and smooth - except over the aluminum where a slight roughness was observed. The surface roughness was probably due to surface roughness of the aluminum substrate produced by an initial powder blast which was performed to obtain adequate adhesion. The inside appeared to be satisfactorily coated.

### 3. Silver Over Electroless Nickel

The original plan was to utilize only silver, but the vendor misread the print. Since the silver adequately covered the electroless nickel, the composite coating was accepted for test. The silver coating appeared uniform in texture and color. The silver was somewhat rough over the aluminum (presumably because the electroless nickel was rough). Internal areas appeared to be adequately covered.

A group of capsules was removed from the furnace after 1000 hours of exposure. The specimens were being evaluated at the close of the report period.

## C. EVALUATION OF BOND AREA OF Al/Cu TRANSITION JOINT

The effect of elevated temperature aging on a Koldweld transition piece (copper/6061 aluminum alloy) was continued. A specimen (Figure 11) which had been exposed at 350°F for 1500 hours showed a slight diffusion zone in the copper (0.00008 in.). For comparison, Figure 12 shows the as-received specimen. No diffusion was detected into the aluminum alloy side of the interface on the aged specimen. Tensile tests on unexposed and exposed specimens were run. The fracture faces of the exposed specimen were metallic gray. A comparison of tensile properties of the joint in the as-received condition (Reference 12) and 350°F aged condition is shown below.

Tensile Properties of 6061-T6 Al/Cu Koldweld Joints

| | Condition | |
|---|---|---|
| | As-Received | Aged 1500 hr at 350°F |
| Ultimate tensile strength, psi | 35,800 | 36,000 |
| Yield strength, psi | 33,150 | - |
| Elongation, % in 2 in. | 21 | 2 |
| Reduction of area, % | 70.4 | 14.4 |
| Point of failure | Aluminum | Al/Cu interface |

The 14% reduction in area of the aged specimen (Figure 13) is judged to indicate adequate ductility for the application. Based upon data generated to date, it appears that the reaction is to strengthen the aluminum alloy by age hardening and thus move the failure area from the aluminum alloy to the joint interface. The test of the aged specimen is the first in which failure in the bond area was produced. Therefore it was deemed necessary to run a longer time test to determine if unacceptable reduction in the ductility of the interface area will occur. Toward this end, thermal exposure at 350°F of a specimen for 3000 hours was started.

A coextruded tubular transition joint specimen of aluminum bonded directly to 304 SS furnished by Nuclear Metals, Inc., Concord, Mass. was examined metallographically in the as-received condition (Figure 14). Another specimen was examined after exposure in air at 275°F for 350 hours (Figure 15). There was no significant diffusion resulting from the elevated-temperature exposure.

## VI. BIMETAL TUBE EVALUATION

### A. 9Cr-1Mo/316 SS BIMETAL TUBE

The objective of this task is to establish the availability of a bimetal tube comprised of nonrefractory, conventional materials. Tubing will be evaluated to establish its acceptability as a backup material to the 9Cr-1Mo steel mercury containment tube for SNAP-8 boiler service.

Ultrasonic inspection of the bond integrity of eight lengths of 9Cr-1Mo/316 SS bimetallic tubing supplied by AGN indicated that four lengths had extensive unbonded areas and four lengths were well bonded. The unbonded tubing was produced by cold drawing of tube blanks which were prepared by inserting a 9Cr-1Mo tube into a 316 SS tube. The bonded tubing was produced by cold drawing of tube blanks which were prepared by hot coextrusion. Sections of the well bonded tubing were machined into specimens for the development of tube-to-tube joining techniques.

Planning was initiated for conducting a thermal shock test on the bimetal bond area. The procedure selected employs heating of the tube OD and water spraying of the tube ID. This method will produce a high $\Delta T$ (100°F) across the tube wall, which simulates boiler operation. A test method utilizing helium was evaluated, but a thermal analysis indicated a helium flow rate of 200 cfh would be required. Due to the large amount of gas required and the associated expense, this procedure was rejected in favor of the water spray method.

### B. REFRACTORY BIMETAL TUBE DEVELOPMENT

The objective of this task is to establish the availability of refractory-bimetal tubing as a backup for mercury containment in the SNAP-8 system if experience indicates that 9Cr-1Mo steel (the present reference material) does not exhibit sufficient mercury-corrosion resistance for a life of 10,000 hours. If direct-bonded tubing (refractory to 316 SS) cannot be fabricated or proves to be unusable because of elevated-temperature diffusion effects, a source is to be developed for refractory-bimetal tubing fabricated using an interface material or materials between the refractory and the 316 SS.

## 1. Tube Materials Diffusion Study

Investigations made on bimetallic tubing have indicated difficulties and potential difficulties in bonding and joining, and in particular the interdiffusion of the refractory liner and stainless-steel cladding materials (Reference 16), for a long-time elevated-temperature application. The result has been the inception of a program to develop bimetal tubing with a diffusion-inhibiting ductile interface. A theoretical literature survey was made to determine probable ductile interface materials. The first phase of the program involved fabrication (by Metals and Controls, Inc., Attleboro, Mass.) and evaluation of flat bond specimens containing candidate interface systems between refractory and 316 SS sheets. These flat bonded specimens and one rod and two tubing specimens (produced by Nuclear Metals, Inc., Concord, Mass.) were exposed to 1350 and 1450°F in vacuum for 500, 1000, and 2000 hours. Pre- and post-exposure metallographic and hardness surveys were made for a first-step evaluation of the systems. The surveys included 112 specimens to make five groups of the following combinations:

Group 1 - Cb/316 SS
Cb/316 SS (tube)
Cb/(1Zr)/316 SS
Cb(1Zr)/316 SS (tube)
Ta/316 SS

Group 2 - Cb/Cu/316 SS

Group 3 - Cb/Fe/316 SS
Cb(1Zr)/Fe/316 SS
Ta/Fe/316 SS

Group 4 - Cb/V/Fe/316 SS
Cb/V/Fe/316 SS (rod)
Cb(1Zr)/V/Fe/316 SS
Ta/V/Fe/316 SS

Group 5 - Cb/Mo/Ni/316 SS
Cb(1Zr)/Mo/Ni/316 SS
Ta/Mo/Ni/316 SS

### a. Exposure Procedure

#### (1) Specimen Preparation

Cutting of exposure specimens from coupons was performed with a hand saw since this method maintained the bond intact.

Average specimen dimensions were 0.25 in. wide by 0.75 in. long.

After cutting, specimens were identified with a code number using a vibrating tool, then degreased in a solvent.

Visual examination of the coupons before cutting revealed that the Ta/Mo/Ni/316 SS specimen was debonded to such a degree that it was difficult to find good bonded areas for test specimens. Specimens cut from this coupon debonded partially during exposure. This sample was not evaluated.

### (2) Test Facility

Vacuum in the test chamber was maintained using a Consolidated Vacuum Corporation Oil Diffusion Pump with water-cooled baffle. The test chamber consisted of a 316 SS tube, approximately 45 in. long with a 4 in. inside diameter. It was provided with necessary connections to the oil diffusion pump, vacuum gages, water cooling coil, and a cover plate with four thermocouple connections.

The vacuum chamber containing the test specimens was installed into an Arcweld sleeve furnace. Temperature control was obtained using Wheelco Temperature Controller.

During preliminary tests with dummy specimens it was found that the required vacuum of $10^{-5}$ mm Hg was readily obtainable at specified temperatures. For vacuum measurements two independent gages were used: (a) discharge vacuum gage type GPH-100A, and (b) thermocouple vacuum gage type GTC-105.

The thermocouple vacuum gage was connected to the furnace power supply; if at any time the vacuum in the test chamber should drop below 40 microns, power to the furnace would be cut off. This arrangement prevented exposure of the test specimens in a vacuum of less than 40 microns. During the actual exposure, pressure in test chamber was always below $10^{-5}$ mm of mercury.

The two required exposure temperatures of 1350°F and 1450°F were obtained in the test chamber, using two heating zones on the sleeve furnace. Regulating the power input to each zone provided uniform temperatures. Temperature was controlled during exposure using one of the four Chromel-Alumel thermocouples welded on to the test specimens and connected to a Wheelco Controller. Two thermocouples were located in the 1450°F zone and two in the 1350°F zone.

Figure 16 shows the specimen stand inside the test chamber and specimen location in the sleeve furnace. To ensure uniform temperature distribution on all specimens, they were positioned tangentially to the wall of the chamber.

Temperature measurements on the remaining three thermocouples were taken twice daily using Leeds & Northrup portable potentiometer No. 8686. The temperature in each zone was maintained well within the required limits of ±10°F.

### (3) Exposure

The vacuum test chamber with the test specimens was installed in the sleeve furnace after the necessary connections had been made. The chamber was then evacuated below $10^{-5}$ mm of mercury. This vacuum, because of the outgassing problem, was maintained at room temperature for 24 hours; then heat was applied slowly to the furnace. The time required to reach steady-state condition was approximately 8 hours. During the 8-hour period, temperature corrections were made in both zones.

After 500 hours of exposure the furnace was shut down. The vacuum in the test chamber was maintained until the temperature inside the chamber indicated 75°F; then some of the specimens were removed. After an additional 500 hours of exposure, another set of specimens was removed (1000 hours exposure). The exposure on the remaining specimens was continued for another 1500 hours (2500-hour exposure).

After completion of the 1000-hour exposure test, the upper portion of the test chamber, as well as thermocouple insulators, was covered with a gray compound. Also, the test specimens in the 1350°F zone were slightly discolored. It was theorized that the sediment was an outgassing product from the thermocouples insulators. The change was made then to quartz-glass insulators.

### b. Evaluation Procedure

The standard columbium etchant (10 cu cm $H_2O$, 5 cu cm $HNO_3$, 2 cu cm HF) was used as an all-purpose etchant and augmented with milder etchants on occasions. Although the Cb etchant was ineffective for Ta, it is believed that it would have etched any diffusion taking place into the Ta.

Knoop hardness indentations were made with a 100-g load and the KHN values converted to Rockwell B or C equivalents. In conversion, some hardness values are probably slightly higher than actual; however, in this survey, relative values are of prime consideration.

### c. Summary of Evaluation

#### (1) Bonds

All of the as-received samples (no heat treatment) showed satisfactory bonding.

#### (2) Diffusion

The diffusion characteristics of the various composites varied from no perceptible diffusion to severe interdiffusion and the formation of intermetallic compounds. There was no difference noted between the diffusion characteristics of Cb and Cb(1Zr). The diffusion characteristics of the various components of the composites can be summarized as follows:

#### (a) Tantalum

Tantalum resisted diffusion of all adjacent composites, except iron, and even at the Ta-Fe interface, diffusion was not conclusive.

### (b) Vanadium

Although vanadium showed excellent bonding and no interdiffusion with Cb, Cb(1Zr), and Ta, it rapidly combined with Fe to form a very hard and brittle compound. In the Cb-V-Fe-SS group, vanadium penetration through the Fe showed a strong influence on the Fe-SS interdiffusion reaction at the longer time and higher temperature heat treatments.

### (c) Copper (1/4-Zr)

The metallurgical bond formed at the Cu-Cb interface appeared to deteriorate rather than diffuse with temperature and time. At the Cu-SS interface, the copper diffused into the SS in an erratic manner resembling corrosion rather than diffusion.

### (d) Molybdenum

A very small amount of diffusion was indicated at the Cb and Cb(1Zr)-Mo interface at high magnification examination. No diffusion with Ta was observed. Molybdenum and nickel rapidly interdiffused.

### (e) Nickel

Ni and Mo rapidly interdiffused. The nickel and stainless steel also rapidly interdiffused, but did not show the customary diffusion line.

### (f) Iron

Iron showed the greatest tendency to diffuse. It also was the most erratic in diffusion behavior. Severe interdiffusion with vanadium and stainless steel was shown and there also was evidence of slight diffusion with Cb, Cb(1Zr), and possibly Ta.

### (g) Cb, Cb(1Zr) Directly to SS

The very thin diffusion layer formed at the Cb and Cb(1Zr)-SS interface showed no appreciable expansion with increased temperature and time. High magnification showed no apparent composition change immediately adjacent to the diffusion layer. The diffusion layer was too thin for hardness measurements, but scratch tests indicated that this is a hard layer. The Cb and especially the Cb(1Zr) immediately behind the diffusion layer shows a band which suggests the possibility of a secondary diffusion layer.

## d. Direct Bonded Refractory to 316 SS Systems

The systems of direct combination of the refractory metal to 316 SS appear to present less of a diffusion problem than was shown by the diffusion barrier materials. Figures 17 through 21 show the effects of the extremes in exposure time at each temperature (1350 and 1450°F) by comparison with unexposed samples. The systems illustrated are Cb, Cb-1Zr and Ta with 316 SS, and include both flat sheet and tube specimens where available.

The Cb showed no structure change or hardness change as the result of exposure (compare Figures 17 and 18). The generally slight hardness increase of the Cb adjacent to Cb-SS junction is believed to be the result of some unknown reaction (other than diffusion).

The stainless structure changes from a severe work-hardened to a "spherodized" (and probably Sigma carbide) condition at 1350°F/500 hr and remains constant thereafter. Carbides show no tendency to diffuse at the interface. The larger and fewer carbides in the SS of the tube specimens indicates a composition variable between these and the sheet specimens. Hardness trends are minor.

The Ta series shows no change at the interface. The Cb series shows a very thin (approximately 0.0001 in.) diffusion zone. This condition is more prominent in the tubing specimens. Only a finite increase in the width of this zone with time and temperature is indicated, although time appears to improve delineation of the zones.

### 2. Tube-to-Header Back-Brazing

The objective of this task is to develop a procedure for producing a welded and back-brazed tube-tube sheet joint. The tube and tube sheet are both constructed of a refractory/316 SS bimetal system. The joint must be so designed that mercury flowing in the tube shall be exposed only to the refractory. Pyromet Co., San Carlos, California, is developing a joining procedure under a SNAP-8 subcontract.

Flat Cb/316 SS bimetal tube sheet specimens were fabricated by Pyromet for back-brazing development. An intermediate layer of iron was used to provide a transition of thermal-expansion coefficients across the bond area. This transition was deemed necessary because of previous unsuccessful attempts to produce a bond between the 0.050-in.-thick Cb and the 0.250-in.-thick 316 SS. A 97.2 Au-2.8 Pd braze alloy was used at the Cb/Fe interface, and a 50 Au-25 Ni braze alloy was used at the Fe/316 SS interface. The layers were braze-bonded using the Horton-Clad Process. This is a procedure where the assembled composite is bonded by exposure to 2000°F while a vacuum is applied to the interfaces of the composite. A cooling rate of 100°F/hour from the bonding temperatures was used to further minimize the possibility of bond separation.

Four sets of machined tube-to-tube sheet joint specimens were prepared by Pyromet and shipped to the Aerojet Von Karman Center. These specimens consist of Cb/316 SS bimetal tubes and the multi-metal tube sheets described above. The joint configuration is shown in Figure 22. These specimens are to be seal-welded on the Cb side by Aerojet-General and then returned to Pyromet for development of the back-brazing technique.

## C. MERCURY CAPSULE TESTS OF BIMETAL TUBE JOINTS

The objective of this task is to determine the mercury corrosion resistance of welded joints in bimetallic 9Cr-1Mo/316 SS, and Cb/316 SS tubing. The tubing was supplied by Nuclear Metals Inc., Concord, Mass., and was fabricated using the hot extrusion cold-drawing process.

Twelve capsules were fabricated for testing as outlined in Table 5. One of each item listed was used as a control for comparison of pre- and post-exposure conditions. The refluxing capsules (Figure 23) were constructed so that one welded joint is below the mercury boiling interface and the second welded joint is in the area where mercury vapor is condensing. All capsules were heated at 1150°F in the SNAP-8 capsule test furnaces described in Reference 13. One set of capsules was removed after 1000 hours exposure, and another set after 2000 hours exposure. Evaluation of the 1000-hour specimens was completed, and evaluation of the 2000-hour specimens was in process, at the end of the report period.

The 1000-hour capsules were opened by sawing off the fill tube, and the mercury was drained off. These capsules were then sectioned and examined to determine the extent of mercury corrosion on the welded joints. Metallographic examination was performed on the upper and lower weld of each capsule. Photomacrographs of each capsule ID were taken as were photomicrographs of the welds.

### 1. 9Cr-1Mo/316 Stainless Steel Bimetallic Capsule (Item 1, Table 5)

The upper and lower welds showed no mercury corrosion even though the lower weld indicated burn-through of the Type 316 stainless-steel cladding weld into the 9Cr-1Mo inner weld (Figure 23).

### 2. 9Cr-1Mo/316 Stainless Steel Bimetallic Capsule (Item 2, Table 5)

The lower weld in liquid mercury showed no mercury corrosion. The upper weld indicated mercury corrosion in two areas at a maximum depth of 0.006 in. Both of the weld areas where corrosion occurred show burn-through of the 316 stainless-steel cladding weld into the 9Cr-1Mo inner weld (Figure 24).

### 3. Cb/316 Stainless Steel Bimetallic Capsule (Item 3, Table 5)

This capsule was electron-beam welded using a controlled penetration procedure such that only the 316 SS cladding was joined.

No mercury corrosion was seen in the upper or lower 316 stainless-steel welds even though the weld penetrated into the columbium liner in some areas (Figure 25), creating a gap at the columbium liner joint.

### 4. 9Cr-1Mo Unclad Capsule (Item 4, Table 5)

The upper and lower welds showed no indication of mercury corrosion (Figure 26). However, corrosion products were noted in the mercury when the capsule was emptied, indicating some corrosion had taken place.

### 5. Discussion

Improvement is needed in the technique for making bimetallic welds in the 9Cr-1Mo/316 stainless steel. All but one weld examined indicated penetration of the 316 stainless steel cladding weld into the 9Cr-1Mo liner weld, resulting in an uncontrolled weld metal composition on the inside surface of the capsule. A small amount of mercury corrosion was seen at these 9Cr-1Mo/316 stainless steel welds in the upper portion of the capsule after 1000 hours at 1150°F. No noticeable corrosion occurred in the lower welds.

The 316 stainless steel weld areas in the columbium/316 stainless steel capsules showed no corrosion after 1000 hours at 1150°F, even though a gap existed at the columbium liner joint. To ensure reliability, it would be desirable to weld the columbium liner when this type of joint is made.

## VII. 9Cr-1Mo STEEL STRENGTH EVALUATION

### A. 9Cr-1Mo WELD STRENGTH

The objective of this task is to evaluate the strength of various weld configurations in the SNAP-8 system where at least one component is 9Cr-1Mo steel. Evaluation will be based on direct comparison of parallel tests of unwelded specimens where applicable.

### 1. 9Cr-1Mo to 9Cr-1Mo Welds

Eight sheet specimens of 9Cr-1Mo material will be tested in air at 1325°F for times up to 3000 hours, as listed below, to evaluate the creep-rupture strength of 9Cr-1Mo to 9Cr-1Mo steel welds.

| | Nominal Stress, psi | | Nominal Stress, psi | |
|---|---|---|---|---|
| | 2300 | | 1600 | |
| Condition | Unwelded | Transverse weld in the center of the gage section | Unwelded | Transverse weld in the center of the gage section |
| Number of Specimens | 2 | 2 | 2 | 2 |

Testing of four of the eight specimens was completed during the report period. The results of the four tests completed are summarized in Table 6 and the creep curve for each specimen is given in Figure 27.

These test results indicate that welding does not decrease the creep or creep rupture strength of 9Cr-1Mo material at 1325°F.

Testing of the remaining four specimens which are duplicates of the four tested specimens was started.

### 2. 9Cr-1Mo to 316 SS Weld Transition Joint

The objective of this task is to evaluate the strength, under simulated SNAP-8 operating conditions, of welded transition joints connecting 9Cr-1Mo steel and 316 SS using 310 SS weld-filler metal. Such a joint is required at various interfaces between the first three loops of the SNAP-8 system. The joint must withstand extended service at a maximum temperature of approximately 1325°F (at the mercury-superheated-vapor manifold of the boiler).

Two welded transition cylindrical specimens (the welds of one specimen had been rolled planished) were installed in a temperature-controlled oven at 1300°F. The exposure tests were conducted concurrently on both specimens using a parallel pressure system operated by a timer arrangement. A pressure switch, burst diaphragm, and temperature relays of the test system were inserted in the controls to prevent damage to the test specimen caused by malfunction. The units were pressure-cycled with nitrogen gas between 48 psig and 52 psig, imposing a cycling stress of 1100 ±44 psi, while the temperature was held constant at 1300°F. The pressure mode and specimen temperatures were monitored on two Speedomax strip charts for 10 min once every 8 hours. The pneumatic system, timer circuits, and controllers performed successfully without incident up to the time of test termination.

The test program was conducted in two phases as indicated below.

a. Simulated System Startup Test

The simulated system startup test involved 25 short-period temperature cycles between ambient and 1300°F. The specimens were placed in the oven, which was preheated to 1300°F and conditioned for 8 hours. Nitrogen gas pressure was cycled every 8 sec during the elevated temperature. The specimens were then removed from the oven and the specimen temperature was allowed to decay over a 16-hour period at atmospheric pressure.

b. Simulated System Service Life Test

In this portion of the test program, a constant temperature was maintained at 1300°F and the pressure cycled every 8 sec.

On 13 March 1964, the two welded cylinders commenced the simulated startup test. When the specimen skin temperature reached 1300°F, the nitrogen gas pressure was applied and cycled between 48 and 52 psig at 8-sec intervals. The test sequence was started at 0800 when the specimens were inserted in the 1300°F oven and removed at 1615 for a 16-hour cool period.

Both specimens completed the 25 temperature cycles without evidence of weld separation or leakage. Examination after the cycle life test revealed scale deposits on the external surfaces and slight expansion of the cylinder heads.

The simulated service life test commenced, utilizing the same specimens that had been subjected to the startup test, on 21 May 1964. After 275 hours, the conditions were interrupted for diametrical measurements and reinstalled after 8 hours. After every 500 hours thereafter, the specimens were remeasured for growth expansion. A total of 554,213 pressure cycles were applied before discontinuing the test program.

The test was terminated after 2227 hours exposure to the simulated life test. Examination of the test capsules after removal from the furnace indicated the following:

> The planished specimen had bulged excessively in the 9Cr-1Mo section (see Figure 28). The wall thickness of this specimen had been significantly reduced by the planishing operation, resulting in a higher imposed stress than applied in the unplanished specimen.
>
> The unplanished specimen was giving no indication of yielding (see Figure 29).

The results of examination of the test specimens and evaluation of the test data are contained in Section VIII,F in Reference 4. Metallographic evaluation of the weld joints was in process at the end of the report period. It is concluded that there is no hazard of failure by creep in a weldment between 9Cr-1Mo and 316 using 310 filler metal. The increases in diameters at the welds can probably be attributed to one, or both, of the following factors: (a) thermal strains due to the difference in coefficient of thermal expansion, and (b) relaxation from the strains due to mismatch at the welds caused by the difference in pipe diameters. This mismatch can be seen in Figure 29 where the OD of the 316 SS pipe (left side in the figure) is slightly larger than the OD of the 9Cr-1Mo pipe.

## B. 9Cr-1Mo ENVIRONMENTAL CYCLIC CREEP

The objective of the cyclic creep test is to determine whether excessive creep of the existing SNAP-8 boilers can be expected, in the desired 10,000-hour lifetime, due to the environmental and operating conditions of the boilers.

It has been established in the SNAP-8 primary loop corrosion program at ORNL that the 9Cr-1Mo boiler tubes will decarburize significantly due to a difference in carbon activity between the various alloys exposed to the NaK. Extreme grain growth has accompanied the decarburization in some cases. Carbon has been removed from a layer up to 0.06 in. deep and to a level as low as 0.002% in 700 hours. The decarburization by itself is not expected to be a serious problem, or to reduce the creep strength of the 9Cr-1Mo to dangerous levels. It has been postulated, however, that the creep strength of newly recrystallized material will be drastically reduced owing to the absence of dislocations, although experimental verification of this phenomenon is not available. Therefore, the average creep strength of the entire wall section may be significantly reduced while recrystallization is actually taking place. If the postulation is true, trouble with future boilers may be avoided by redesign. However, existing boilers present a more urgent problem.

It was also established in the ORNL program that decarburization proceeds much more slowly in static NaK than under dynamic loop conditions, as if the rate of carbon diffusion through the liquid phase were the controlling factor. Therefore, an electromagnetic stirring method was devised for the creep test.

Since it is imperative that the test be conducted under conditions that will produce decarburization and grain growth, a trial run was devised to establish the validity of the test design.

### 1. Trial Run

The objective of the trial capsule run was to verify that the conditions for the NaK cyclic creep test would result in decarburization and grain growth of the 9Cr-1Mo creep specimens.

### a. Equipment Design

A drawing of the capsule is shown as Figure 30. Two 1-1/4 in. Schedule 5, 316 stainless steel weld caps welded end-to-end were used as the capsule. A 2-in. length of 9Cr-1Mo boiler tube, conforming to ASTM-A199-61T, was used as a specimen.* A cap was welded to the bottom of the 9Cr-1Mo tube specimen to minimize circulation of the NaK from the outside to the inside of the specimen. Thus, decarburization of the 9Cr-1Mo in NaK exposed to the 316 stainless steel and in NaK exposed only to the 9Cr-1Mo can be compared. Some circulation will occur through the slots at the top of the specimen. The slots aid in maintaining a proper NaK level on each side of the 9Cr-1Mo specimen. Isolated NaK expansion on either side during capsule heat-up might otherwise result in an unsatisfactory liquid inventory. The tabs (three equally spaced around the specimen circumference between these slots) are bent over to center the specimen in the 316 SS tube.

The capsule was mounted in a furnace block with three stirring coils wound lengthwise around the capsule. These coils (120° apart) were hooked up in 3-phase delta so that a moving magnetic field would stir the NaK. Chromel-Alumel thermocouple wire was used for the coils since it was the only readily available wire with high-temperature insulation. A field of about 72 ampere-turns was selected to limit heat generation in each coil to about 50 watts.

### b. Operation

The capsule and parts were cleaned by standard SNAP-8 boiler cleaning procedures (Reference 14). The specimen was weighed. The capsule was loaded with NaK. A quantity was transferred in a vacuum glove box into a 316 stainless-steel funnel with a sintered powder filter ($5\mu$ opening) in the bottom. Argon was admitted to the box and a hypodermic needle was loaded with NaK filtered at room temperature. After the hypodermic needle was washed several times with filtered NaK, a measured amount of NaK was inserted into the capsule. The holes in the capsule were then welded shut, trapping argon above the NaK.

The stirring coils were wrapped around the capsule so that they would fit into the mild steel positioning block. Preliminary tests were made to adjust the voltage across each coil so that the fields were nearly equal and only about 50 watts were generated in each coil.

The capsule was inserted into a capsule furnace and the run started on 9 October. Operating temperature was 1325°F as measured by two thermocouples - one next to the capsule and one next to the block. After 500 hours the capsule was removed from the furnace for evaluation.

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*Material was from a lot purchased for single-tube boiler test sections. Material from the lot used to build existing RPL-2, PCS-1, PCS-2, and LeRC boilers was not available.

c. Results

The capsule was evaluated using the following procedures: (1) cut open top end of capsule and drain excess NaK; (2) wash out remaining NaK with alcohol; (3) remove specimen, examine visually, and weigh; (4) cut specimens for metallographic analysis from the 316 stainless-steel capsule wall and the specimen wall; and (5) determine degree of decarburization and grain growth.

Visual examination disclosed no indication of carbon transfer or corrosion.

Micrographs revealed both decarburization and grain growth in the 9Cr-1Mo specimen (Figure 30). A layer about 0.006-0.007 in. shows freedom from precipitated carbides and increase in the average ASTM grain size from 6-1/2 to 5. A slight increase in carbon content at the exposed surface of the 316 SS could be discerned visually from a microscope but does not show on the photograph of the microstructure (Figure 30).

It was concluded that the 9Cr-1Mo lot would decarburize and increase in grain size as desired under the conditions of the cyclic creep test.

## 2. Cycle Creep Test

a. Experimental Approach

Three 9Cr-1Mo capsules will be stressed in a NaK environment at 1325°F to simulate the SNAP-8 boiler environment. Stresses will be induced by pressurizing the internal NaK until the desired biaxial stress exists in the tube wall. Thin sections will be machined in the wall to control the location of the stress and to permit two different stresses to be investigated in each capsule. The pressure will be changed each 30 sec so that a ±10% stress cycle results. Temperature will not be cycled because its effect can be predicted.

The three capsules will be immersed in NaK inside a Type 316 stainless-steel capsule and the assembly operated in the capsule creep furnace. It is expected that the concurrent exposure of the 316 stainless steel and 9Cr-1Mo in NaK will result in transfer of carbon from the 9Cr-1Mo to the 316 stainless steel.

All three capsules will be connected to a common pressurized argon system and, therefore, to the same pressure cycle. The test will start with all capsules under stress. The test will be shut down after 1000, 1800, and 3000 hours. During each shutdown, the pressure will be released from one capsule and the pressure system disconnected from the capsule. Wall thicknesses of the gage sections will be selected in each capsule so that one section will creep an estimated 1% and the other will creep an amount estimated to be equivalent to that of the SNAP-8 -1 model boiler. Therefore, if detrimental effects due to the stress cycling, biaxial stress, NaK environment, or C transfer occur, they can be readily determined by comparing the actual creep to that predicted.

### b. Design

The capsule creep method was selected for this test because it accurately simulates the stresses and environmental conditions of the SNAP-8 -1 boiler and because of the availability of apparatus.

In the boiler, pressure is exerted by the mercury. However, in this test, pressurized NaK will be used because it was believed that mercury was not a variable in the problem and its use requires complicating the test design.

The apparatus consists of a capsule assembly, which includes the creep capsules, outer capsule, and vapor traps; a furnace assembly, which includes the capsule assembly, the furnace, and the furnace stand; and a gas pressurizing (GP) system, which includes the gas purifier, the pressure regulators, valves, gages, and safety shutdown system. The design of the test system is covered in Section VIII,I in Reference 4.

## VIII. CORROSION LOOP PROGRAM

The objectives of the Corrosion Loop Program are (a) to determine corrosion and mass-transfer patterns in the mercury and NaK loops of the SNAP-8 system, (b) to evaluate the corrosion resistance of the SNAP-8 reference materials against the 10,000-hour-life requirement, and (c) to develop and test mercury corrosion product separators that will remove solid particles from the liquid mercury and superheated mercury vapor. The program is being conducted at Aerojet-General Nucleonics (AGN), San Ramon, California.

Component Test Loop 2 (CTL-2), constructed of Haynes 25 alloy, is being operated to check the performance of certain components to be used in Corrosion Loops 3 and 4 (CL-3 and -4), which are being constructed to 1/16th scale of the SNAP-8 system. The NaK primary loop of CL-3 and CL-4 will be constructed of 316 SS, with chromized Hastelloy N in the high-temperature area and a section of Hastelloy C in the low-temperature area. The presence of the latter two alloys is dictated by the use of these materials in the reactor of the SNAP-8 system.

### A. CORROSION LOOP 4

### 1. Hydrogen Injector System

In the SNAP-8 reactor, hydrogen is released from the zirconium-uranium hydride fuel elements and diffuses into the NaK coolant. The hydrogen can diffuse from the NaK system to space or through the boiler into the mercury system where it will accumulate. The effects of this incondensable hydrogen gas collected in the mercury system will be investigated in CL-4. In particular, it is expected that the heat-transfer characteristics of the boiler and condenser will be affected.

The hydrogen injection rate selected for CL-4 (8.13 cu cm/hour STP) was based on the ratio of the 9Cr-1Mo mercury boiler tube area in the corrosion loop to the area of the 9Cr-1Mo boiler tubes in the reference SNAP-8 system. Other scaling factors between the reference SNAP-8 system and the corrosion loops that were considered are given at the top of the following page.

| Parameter | $H_2$ Injection Rate into the Primary NaK System of CL-4 cu cm/hour (STP) |
|---|---|
| Mercury flow rate | 2.61 |
| Mercury condenser surface area | 3.5 |

The highest injection rate of 8.13 cu cm/hour was selected because with this rate the effects of hydrogen on the operation of the mercury system should become more quickly apparent. The injection rate of the hydrogen injection system is flexible enough so that the range of injection rates listed above can be accommodated.

The hydrogen injector system basically consists of a hydrogen bottle, a hydrogen purifier, and an injection tip located in the primary NaK loop between the heater and the boiler. The injection tip is shown in Figure 31 and a schematic of the system is shown in Figure 32.

Bottled hydrogen is reduced to 200 psig, and passed through a palladium diaphragm-type hydrogen purifier. The hydrogen flows through a coil of small-diameter tubing to limit the flow in case of a failure in the system. The hydrogen then flows to the injector tip where it diffuses through the stainless-steel tubing into the NaK.

The injection rate is controlled by the hydrogen pressure at the injector tip. A pressure transducer is installed near the injector tip so that the injection rate can be checked periodically. This is done by holding a known volume of hydrogen behind the injector tip and monitoring the pressure decay. The loss in pressure vs time indicates the rate of diffusion of hydrogen through the tip into the NaK.

Ordering of materials and components for the hydrogen injector system was started.

### 2. Component Fabrication

The fabrication of the liquid corrosion product separator for CL-4, described in Section VIII,B,2 in Reference 1, was completed. The material used in construction of the liquid separator was 9Cr-1Mo, except for the columbium and iron wool.

### 3. Loop Assembly

The assembly and the installation of CL-4 was completed during the report period checkout of the instrumentation, and electrical hookup was started. The following modifications were made during the assembly:

a. The hot trap in the NaK purification system was not installed since experience with CL-3 indicated that the oxide level in the NaK could be controlled with the cold trap.

b. An Hg liquid corrosion product separator was installed in the loop on a parallel line containing isolation valves. Its use is not anticipated. Installation now, however, eliminates test schedule slippage in the future if the separator is required.

c. The turbine simulator valve between the blade mockup and the condenser inlet was not installed since the operation of CL-3 showed the valve was not necessary.

d. The boiler plug was removed and the pitch of the wire turbulator along the plug was changed from 1.5 in. to 0.75 in. Before the plug was reinstalled in the boiler, the 9Cr-1Mo tubing in the boiler was cleaned by filling the coil with Freon (PCA)* and draining it. Argon was then blown through the coil to remove excess Freon.

e. An immersion thermocouple was installed at the boiler outlet so that the mercury vapor temperature could be monitored more accurately.

## B. CL-3 OPERATION

### 1. NaK Systems

The primary and the condensing NaK systems of CL-3 operated satisfactorily up until the final plugging run was being made prior to loop shutdown on 23 December. During this plugging run a leak developed in the vicinity of the NaK heater. The plugging run could not be completed, so the final oxide content of the NaK in the primary system prior to shutdown could not be determined.

The history of the oxide control in the primary loop is given below.

| Operation Time (hours) | Oxide Level (ppm) | Remarks |
|---|---|---|
| 100 | 38 | Cold trapped (250°F) for 4 hours; oxide level reduced to 18 ppm |
| 530 | 20 | |
| 1130 | <20 | |
| 2100 | 38 | Cold trapped (225°F) for 5 hours; oxide level reduced to 18 ppm |
| 3200 | <25 | |

Preliminary examination of the NaK primary system indicated the damage from the NaK leak was minor and the corrosion results from the loop would not be affected.

### 2. Mercury System

The operating condition of the mercury system during this report period is represented by Figure 33. Shown are a daily plot of the corrected mercury boiler outlet temperature, the mercury saturation temperature that corresponds to the boiler outlet pressure, the mercury flow rate, and the effective flow area of the choked nozzle. The corrected mercury boiler outlet temperature is the sum of the boiler outlet skin thermocouple readings plus the calculated temperature drop across the mercury vapor film. The temperature gradient across the vapor film is about 57°F (see Appendix C in Reference 4).

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*Product of du Pont Co. (PCA Precision Cleaning Agent).

Mercury boiling was continuous from 19 June through 28 August. The system was operated continuously for 1650 hours except for a few momentary shutdowns resulting from electrical interruption. Throughout the run the boiler outlet temperature was maintained at about 1260°F (corrected). The flow rate was varied between 490 and 530 lb/hour. The choked nozzle was adjusted so that the boiler outlet pressure was maintained at 260 to 270 psia.

The loop was shut down intentionally on 28 August for maintenance. During the shutdown the following items were completed:

a. The water coolant lines (including the cooling jackets of the mercury pumps) were flushed

b. A new D/P cell for the mercury flowmeter was installed to replace the inoperative cell

c. Defective thermocouples on the condenser were replaced

d. Thermal insulation was added to the condensing NaK loop and sections of the mercury loop between the condenser outlet and the pump inlet to correct an excess cooling capacity

e. The condenser and the lines connecting the condenser to the pump were examined by X-rays for possible corrosion and mass transfer.

The loop was restarted on 8 September, and a mercury leak developed on the low pressure connection of the D/P cell for the mercury flowmeter. The loop was shut down and the mercury leak was repaired and checked out.

The loop was restarted on 10 September. Superheated mercury was generated immediately after mercury injection. Four hours after loop startup, a mercury flow rate of 480 lb/hour was achieved with a boiler outlet temperature of 1190°F at 95 psia. The boiler outlet pressure was then gradually increased by closing the choked nozzle. Sixteen hours after startup the boiler outlet conditions were 1260°F (corrected) and 260 psia at a mercury flow rate of 500 lb/hour.

The loop was shut down on 16 September when a slight mercury leak was detected around the boiler outlet. It was found that the leak was caused by the failure of a section of 1/8-in. OD tubing that joined the pressure transducer to the boiler outlet tubing. The tubing was replaced and the loop was restarted on 18 September. Superheat was achieved immediately on startup. Eighteen hours after startup, the boiler outlet conditions were 1260°F (corrected) and 265 psia at a mercury flow rate of 500 lb/hour.

The loop operated continuously for about 750 hours until 20 October, when a 6-hour shutdown was caused by failure of the pump in the plant cooling water system. During the shutdown, the operating of the choked nozzle was checked. The operating mechanism of the choked nozzle was difficult to turn when first operated. After that, the adjustment seemed to be normal again. The loop was restarted, and superheated mercury was produced immediately. The boiler outlet temperature was at about 1260°F (corrected) and mercury flow at 510 lb/hour some 3 hours after mercury injection. However, the boiler outlet pressure increased to about 280 psia, and increasing the choked nozzle opening did not lower the pressure. Evidently, the nozzle mechanism was inoperative. The mercury flow rate was then adjusted to approximately 490 lb/hour in order to maintain the boiler outlet pressure at 275 psia. Since the choked nozzle was not operable, the mercury flow rate was used to control the boiler outlet pressure.

Loop operation continued through 28 October when the loop was intentionally shut down so that modifications to the automatic controllers could be made.

Loop operation was resumed on 4 November and continued through 23 December when the NaK leak occurred in the primary NaK system. The total mercury boiling time for CL-3 was 4400 hours. Preparations were started for dismantling of the loop and evaluation of components and pipe sections.

### 3. Discussion

The boiler performance was improving with increased operation time as indicated by a change in the NaK shell temperature profile slope. Two temperature profiles are shown in Figure 34. The superheat length was about 15 ft on 4 August, and on 22 October the superheat length was about 30 ft. The operating time interval between these two dates was 1530 hours. As the superheat length increased, the pressure drop across the boiler increased from 45 to 65 psi.

The typical NaK shell temperature profile of the mercury condenser is shown in Figure 35. The mercury vapor entered the condenser at 680°F and subcooled to 500°F at the exit.

The reason for the failure of the choked nozzle could not be determined without removing the nozzle from the loop. The operating mechanism that moves the pintle appeared to function properly. It was tentatively concluded that the pintle had become disconnected from the operating mechanism, fixing the nozzle opening.

### 4. Evaluation

The X-rays of the CL-3 mercury condenser and tubing running from the condenser outlet to the pump inlets were evaluated. No corrosion or mass transfer deposits could be seen in any of the X-ray films.

The failed boiler outlet transducer leg in CL-3 was examined. The transducer leg was a piece of 1/8-in. OD by 0.015 in.-wall Type 316 stainless-steel tubing. The tubing failed near the weld where the small tubing was welded into the 9Cr-1Mo boiler outlet tubing. A section of the weld and tubing from the area where the failure occurred was examined metallographically. The failure was caused by mercury corrosion of the 316 stainless-steel tubing near the area where the stainless-steel tubing was welded to the 9Cr-1Mo tubing. The temperature of the mercury vapor stream in the area of the failure was 1265/1250°F. It was also observed that the wall thickness of the 1/8-in. OD 316 SS tubing had been reduced during the welding.

## C. CORROSION PRODUCT SEPARATOR

Component Test Loop 2 was operated after the installation of a new Haynes 25 boiler so that vapor-phase corrosion product separators could be tested. Operation of CTL-2 for this purpose is described below. Operating conditions for the various runs are given in Table 7.

### 1. Run 1

The object of this run was to obtain base conditions for testing vapor corrosion product separators. No vapor corrosion product separator was installed since the significant result of this run was expected to be the time required for the outlet pressure of the boiler to rise from 250 to 300 psig as a result of corrosion product buildup in the adjustable nozzle.

Before the mercury flow was started, the boiler was outgassed at 1100°F for 66 hours. The vacuum reading at the loop was less than 1 micron. When mercury flow was started, superheated mercury vapor was obtained immediately and a boiler outlet temperature of 1265°F at 250 psig was achieved. The loop was operated for 48 hours before shutdown. No buildup of boiler outlet pressure occurred, indicating that corrosion products did not build up in the nozzle as was expected from previous operating experience. The nozzle was removed from the loop, cleaned, and rewelded into the loop. No indication of corrosion product buildup was found on the nozzle.

### 2. Run 2

Since corrosion product buildup did not occur on the nozzle during Run 1 at rated conditions, it was decided to lower the NaK temperature in the CTL-2 boiler to see if a lower degree of superheat would cause buildup in the nozzle.

The loop was started and the boiler outlet pressure was adjusted to 250 psig. The NaK temperature was decreased so that the mercury vapor outlet temperature was approximately the same as the saturation temperature. Under these operating conditions the boiler outlet pressure increased from 250 to 300 psig in 16.7 hours, indicating that buildup of corrosion products in the nozzle had taken place (Table 7). The loop was shut down and the nozzle was removed and disassembled. Corrosion products were found on the pintle and the venturi throat of the nozzle. The nozzle parts were cleaned to remove the corrosion products; the nozzle was assembled and welded into the loop.

### 3. Run 3

The object of this run was to test the effectiveness of an impingement type vapor-phase corrosion product separator. The impingement separator was installed in CTL-2 between the boiler outlet and nozzle inlet as described in Section VIII,B,1 in Reference 15.

The loop was started, using the procedure followed in Run 2. The boiler outlet pressure increased from 250 to 300 psig in 10 hours, indicating that the impingement separator was not effective in preventing corrosion product buildup in the nozzle. The $\Delta P$ across the separator was 20 psi during the 10-hour test (Table 7).

The nozzle and separator were removed from the loop for examination. The nozzle pintle and venturi throat showed corrosion product buildup. Visual examination of the screens from the impingement separator indicated that some corrosion products had been trapped by the screens. A more detailed examination of the separator screens and an analysis of the deposits in the screen were started.

### 4. Discussion

The operation of CTL-2 for the corrosion product separator runs has indicated that the vapor separators may not be needed if the boiler is operating satisfactorily. As shown in Runs 1 and 2 (Table 7), when the boiler produced superheated mercury vapor at rated SNAP-8 conditions, no buildup of corrosion products occurred on the nozzle. However, when the NaK temperature in the boiler was reduced and the degree of superheat was reduced to a low value, buildup occurred on the nozzle in a short time.

Although the temperature profile along the mercury boiler coil in CTL-2 cannot be monitored, it is assumed that the reduction of the NaK temperature in the boiler will shorten or eliminate the superheat length at the outlet end of the mercury boiler coil. This will result in more liquid carryover in the vapor since there is less area in the boiler coil for drying the vapor. The liquid carryover with dissolved corrosion products could then be assumed to be the source of the corrosion products in the nozzle.

In forced convection mercury boiler design, the boiler coil should be long enough so that liquid carryover with the mercury vapor is reduced to a minimum. This may eliminate the need for a corrosion product separator in the vapor area of the SNAP-8 system.

The impingement separator tested in CTL-2 was designed to remove mercury droplets 0.5 microns in diameter with 99% efficiency (Section VIII,B,1 in Reference 15). Since corrosion product buildup occurred when the separator was installed, the liquid carryover droplet size must be less than 0.5 microns. This conclusion is based on the assumption that liquid carryover causes deposit of corrosion products in the nozzle.

## REFERENCES

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4. SNAP-8 Electrical Generating System Development Program, Aerojet-General Corporation Report No. 0390-04-18, January 1965.

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13. SNAP-8 Topical Materials Report for 1962, Vol. III - SNAP-8 Mercury Corrosion Materials Research, Aerojet-General Corp. Report No. 2517, August 1963.

14. Cleaning, Handling and Storage of 9Cr-1Mo Steel Parts, Aerojet-General Corporation Specification No. AGC-60248.

15. SNAP-8 Electrical Generating System Development Program, Aerojet-General Corporation Report No. 0390-04-15 (Quarterly), March 1964.

16. Mercury Corrosion Loop Testing Program, Aerojet-General Corporation Final Report No. 0584, August 1963.

## TABLE 1

## STRESS-RELIEVING TEMPERATURE EFFECTS ON WELDED 9Cr-1Mo AND WELDED AISI 4340

| Material | Stress Relief Treatment | Weld Hardness Rockwell C | Parent Metal Hardness Rockwell B | Flattening Test Results (Distance Between Plates at Failure, in.*) | |
|---|---|---|---|---|---|
| | | | | Weld Bead On | Weld Bead Removed |
| 9Cr-1Mo | None (as welded) | 45 | 81 | 0.5 | 0.688 |
| 9Cr-1Mo | 400°F – 16 hr | 43 | - | - | - |
| 9Cr-1Mo | 1225°F - 1 hr | 25 | 77 | 0.375 | 0.437 |
| 9Cr-1Mo | 1350°F - 1 hr | 21 | 77 | 0.375 | 0.312 |
| AISI 4340 | None (as welded) | 55 | 100 | - | - |
| AISI 4340 | 1225°F - 1 hr | 32 | 90 | - | - |
| AISI 4340 | 1275°F - 1 hr | 31 | 91 | - | - |
| AISI 4340 | 1350°F - 1 hr | 28 | 91 | - | - |
| AISI 4340 | 1400°F - 1 hr | 34 | 101 | - | - |

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*Flattening test performed in accordance with ASTM A-450, with weld located 90° from application of load. This specification dictates that at failure, the distance between plates of ferritic steels must be 0.623 in. or less.

## TABLE 2

## BOILER CONDITIONING TESTS SUMMARY OF CTL-2 OPERATION

| | Run A<br>(Base Run) | Run B<br>(Silicone Oil Addition) | Run C<br>(Run B Continued, Lower Pressure in Boiler*) | Run D<br>(Alkaline Cleaning) | Run E<br>(Run D Continued) | Run F<br>(Run E Continued, Lower Pressure in Boiler*) | Run G<br>(Repeat of Run B) | Run H<br>(Repeat of Runs D and E) | Run I<br>(Restart After Run H) |
|---|---|---|---|---|---|---|---|---|---|
| Mercury Flow Rate, lb/hr | | | | | | | | | |
| By Flowmeter | 1185 | 1170 | 1170 | 1170 | 1160 | 1170 | 1160 | 1170 | 1160 |
| By Power Input | 1040 | - | - | - | 1090 | 1090 | - | 1089 | 1059 |
| Average Power Input, kw | 45.9 | 20.2 | 20.3 | 15.2 | 48.6 | 48.9 | 15.3 | 49.4 | 48.0 |
| NaK Temperature, $^{o}F$ (Control Thermocouple) | 1260 | 1260 | 1260 | 1260 | 1260 | 1260 | 1260 | 1250 | 1250 |
| Boiler | | | | | | | | | |
| Inlet Temperature, $^{o}F$ | 490 | 440 | 380 | 495 | 495 | 525 | 450 | 450 | 460 |
| Inlet Pressure, psia | 330 | 194 | 197 | 155 | 355 | 335 | 147 | 307 | 303 |
| Outlet Pressure, psia | 300 | 175 | 173 | 133 | 310 | 300 | 128 | 267 | 257 |
| $\Delta P$ Across Boiler, psi | 30 | 19 | 24 | 22 | 45 | 35 | 19 | 40 | 46 |
| Outlet Temperature, $^{o}F$ | 1298 | 1060 | 1060 | 970 | 1215 | 1252 | 932 | 1240 | 1260 |
| Boiler Outlet Sat. Temp, $^{o}F$ | 1090 | 995 | 993 | 968 | 1103 | 1090 | 948 | 1068 | 1062 |
| Estimated Vapor Quality | Superheated 208$^{o}F$ | 26.8% | 28.9% | 20.6% | Superheated 112$^{o}F$ | Superheated 162$^{o}F$ | 22.3% | Superheated 182$^{o}F$ | Superheated 198$^{o}F$ |
| Operating Time, hr | 12 | 24 | 26 | 3 | 25 | 13 | 12 | 53 | 20 |
| Time Required to Reach Rated Boiler Outlet Pressure (265 psia), hr | 1.4 | - | - | - | 12.5 | 0.75 | - | 48 | 0.75 |

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*During the pre-run outgassing period.

## TABLE 3

## COMPARISON OF PROPERTIES OF MIX-4P3E* AFTER TESTS WITH LUBRICANT/COOLANT PUMP-MOTOR ASSEMBLY

| Test | Original | After 510 hr L/C PMA Operation | After 1000 hr L/C PMA Operation |
|---|---|---|---|
| Viscosity, centistokes | | | |
| at 75°F | 205.5 | 197.2 | 181.1 |
| at 210°F | 6.31 | 6.14 | 6.24 |
| Volatile content, %<br>(by gas chromatography | None<br>(probably less than 0.01) | Trace<br>(probably less than 0.1) | 0.42 |

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*Mix-4P3E utilized for these tests was from Lot 43-2 of Dow Chemical Company's ET-378 material.

## TABLE 4

## POTENTIAL ALTERNATIVE LUBRICANT/COOLANT FLUIDS FOR SNAP-8 SERVICE

| Fluid Type | Manufacturer | Manufacturer's Designation | MLO Designation |
|---|---|---|---|
| Alkyl diphenyl ether | California Research Corporation | Calresearch 216 | - |
| Alkyl disiloxane | Standard Oil of California | Oronite 70 | - |
| Aromatic hydrocarbon (synthetic) | Socony Mobil Oil Co. | XRM-100 | - |
| Di-(2-ethylhexyl)sebacate | Rohm and Haas | Plexol-201 | - |
| Naphthenic mineral oil, deep-dewaxed, super-refined | Bray Oil Co. | Brayco 777 | MLO 7277 |
| | Humble Oil Co. | Humble 3158 | MLO 7277 |
| Naphthenic white oil | California Research Corporation | Naphthenic white oil | - |
| Neopentyl ester | | | |
| Pentaerythritol tetraheptonoate | Hercules Powder Co. | Hercoflex 600 | - |
| Pentaerythritol tetranonate | Bray Oil Company | Brayco 830 | - |
| Trimethyolpropane ester | Hayden-Newport Co. | Pentalube TP 653 | - |
| Paraffinic mineral oil, super-refined, deep-dewaxed | Humble Oil Co. | Humble 3160 | MLO 60-294 |

## TABLE 5

## MERCURY REFLUX CAPSULES

| Item | No. of Capsules | Material | Fabricated by | Method of Welding |
|---|---|---|---|---|
| 1 | 3 | 9Cr-1Mo/316 SS bimetallic tubing | SNAP-8 Division, Von Karman Center | TIG (tungsten inert gas) |
| 2 | 3 | 9Cr-1Mo/316 SS | AGN | TIG |
| 3 | 3 | Cb/316 SS | Airite Products, Los Angeles, Calif. | Electron beam |
| 4 | 3 | 9Cr-1Mo Unclad | AGN | TIG |

## TABLE 6

## RESULTS OF 9Cr-1Mo CREEP SPECIMEN TESTS

| | Nominal Stress, psi | | | |
|---|---|---|---|---|
| | 2300 | | 1600 | |
| Condition | Welded | Unwelded | Welded | Unwelded |
| Sample No. | 1 | 6 | 2 | 5 |
| Actual Stress, psi* | 2285 | 2333 | 1628 | 1591 |
| Gage Length, in. | 1.567 | 1.568 | 1.555 | 1.566 |
| Elongation, in. | — | — | 0.034 | 0.039 |
| Time to Rupture, hr | 1445 | 1070 | Did not rupture | |

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*Based upon minimum specimen dimension.

## TABLE 7

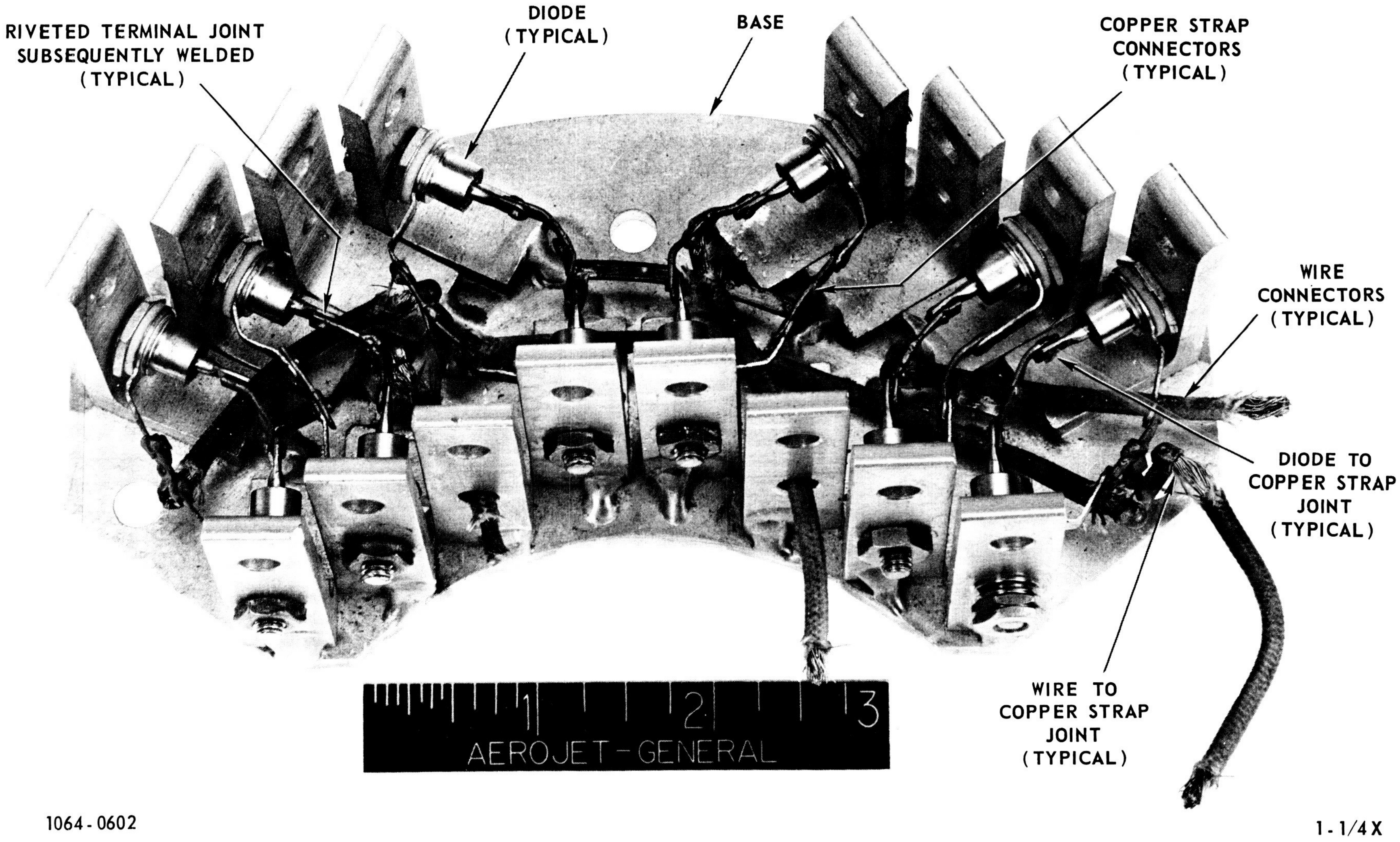
### OPERATING DATA FROM COMPONENT TEST LOOP 2 DURING VAPOR-PHASE CORROSION-PRODUCT SEPARATOR TESTING

| Elapsed Time from Start of Run hr | Mercury Flow Rate lb/hr | Boiler Inlet Pressure psig | Boiler Outlet Pressure psig | Boiler Outlet Temperature °F | Impingement Separator Inlet Temp °F | Impingement Separator Outlet Temp °F | ΔP Across Impingement Separator psi |
|---|---|---|---|---|---|---|---|
| Run 1 Base Run | | | | | | | |
| 4 | 1130 | 312 | 255 | 1240 | | | |
| 8 | ↓ | 305 | 255 | 1290 | | | |
| 12 | ↓ | 288 | 235 | 1280 | | | |
| 16 | ↓ | 273 | 222 | 1255 | | | |
| 20 | ↓ | 273 | 225 | 1325 | | | |
| 24 | 1130 | 277 | 232 | 1270 | | | |
| 28 | 1175 | 310 | 265 | 1265 | | | |
| 32 | ↓ | 310 | 265 | 1260 | | | |
| 36 | ↓ | 310 | 265 | 1280 | | | |
| 40 | ↓ | 308 | 265 | 1265 | | | |
| 44 | ↓ | 312 | 267 | 1280 | | | |
| 48 | 1175 | 312 | 268 | 1298 | | | |
| Run 2 Base Run — Reduced NaK Temperature in Boiler | | | | | | | |
| 5 | 1140 | 320 | 250 | 1125 | | | |
| 8 | ↓ | 305 | 255 | 1125 | | | |
| 10 | ↓ | 315 | 260 | 1140 | | | |
| 12 | ↓ | 320 | 265 | 1105 | | | |
| 14 | 1140 | 325 | 270 | 1095 | | | |

## TABLE 7 (cont.)

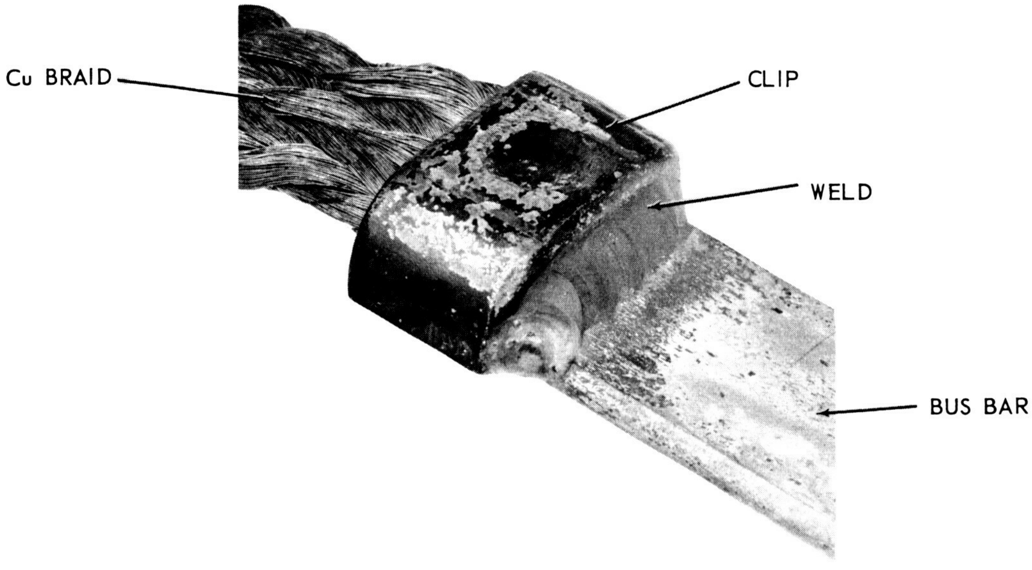
| Elapsed Time from Start of Run hr | Mercury Flow Rate lb/hr | Boiler Inlet Pressure psig | Boiler Outlet Pressure psig | Boiler Outlet Temperature °F | Impingement Separator Inlet Temp °F | Impingement Separator Outlet Temp °F | ΔP Across Impingement Separator psi |
|---|---|---|---|---|---|---|---|
| Run 2 Base Run — Reduced NaK Temperature in Boiler (cont.) | | | | | | | |
| 16 | 1140 | 325 | 270 | 1115 | | | |
| 18 | ↓ | 330 | 275 | 1125 | | | |
| 20 | | 335 | 280 | 1090 | | | |
| 22 | | 340 | 285 | 1065 | | | |
| 24 | 1140 | 370 | 320 | 1060 | | | |
| Run 3 — Impingement Separator Test | | | | | | | |
| 1.5 | 1140 | 325 | 265 | 1060 | 1060 | 1060 | 20 |
| 3.5 | ↓ | 335 | 270 | 1060 | 1070 | 1070 | 20 |
| 5.0 | | 340 | 280 | 1065 | 1070 | 1070 | 20 |
| 6.0 | | 350 | 285 | 1065 | 1075 | 1075 | 18 |
| 8.0 | | 355 | 290 | 1075 | 1080 | 1080 | 20 |
| 10.0 | | 365 | 300 | 1075 | 1080 | 1080 | 18 |
| 12.0 | 1140 | 360 | 300 | 1080 | 1080 | 1080 | 18 |

1064-060

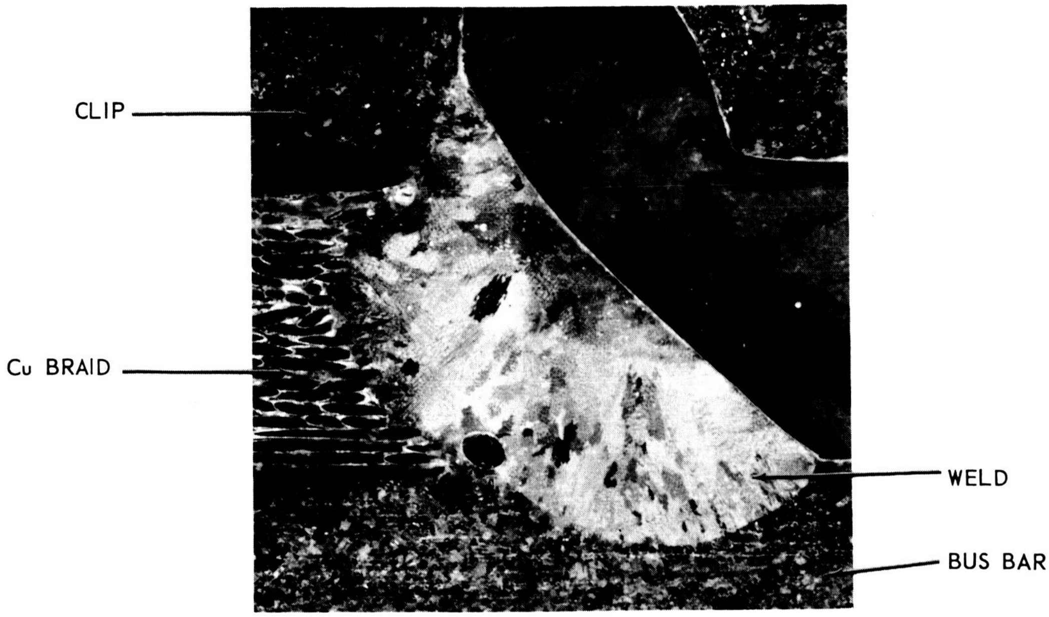

RIVETED TERMINAL JOINT SUBSEQUENTLY WELDED (TYPICAL)
DIODE (TYPICAL)
BASE
COPPER STRAP CONNECTORS (TYPICAL)
WIRE CONNECTORS (TYPICAL)
DIODE TO COPPER STRAP JOINT (TYPICAL)
WIRE TO COPPER STRAP JOINT (TYPICAL)
1
2
3
AEROJET-GENERAL
1064-0602
1-1/4X

Lower Level of Speed Control Module

Figure 1


Cu BRAID
CLIP
WELD
BUS BAR
L-7807
2X

A. JOINT CONFIGURATION

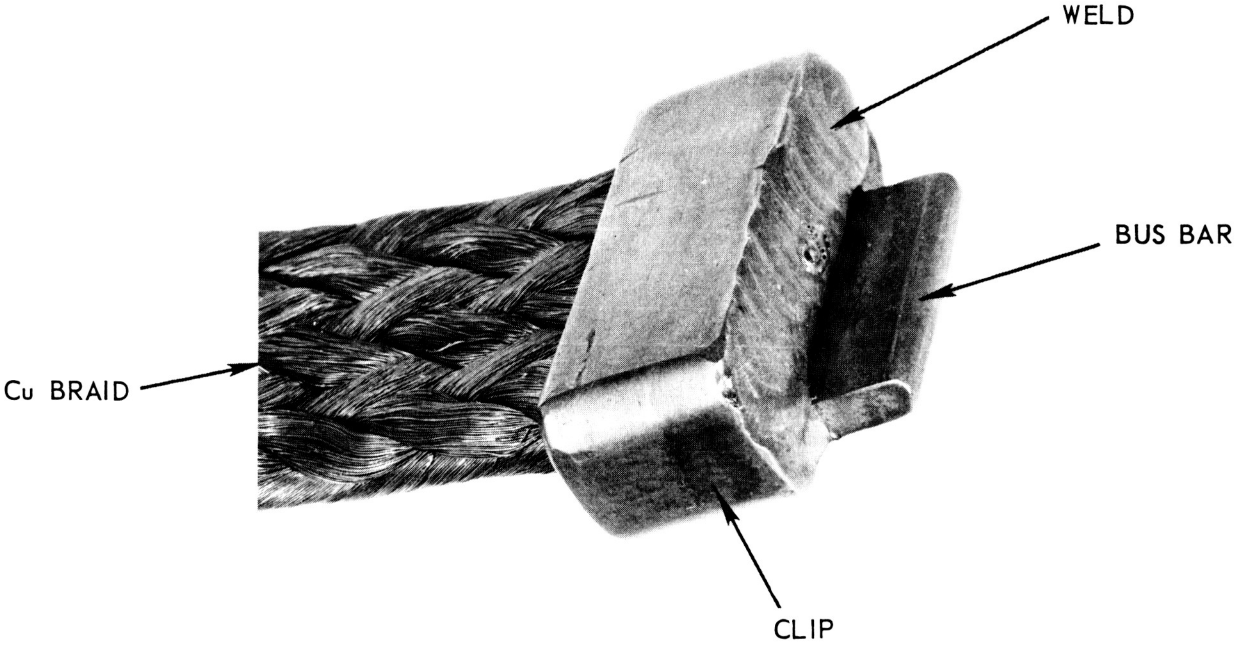

CLIP
Cu BRAID
WELD
BUS BAR
L-7809
15X

ETCHANT –
75% $HNO_3$ + 25% ACETIC ACID

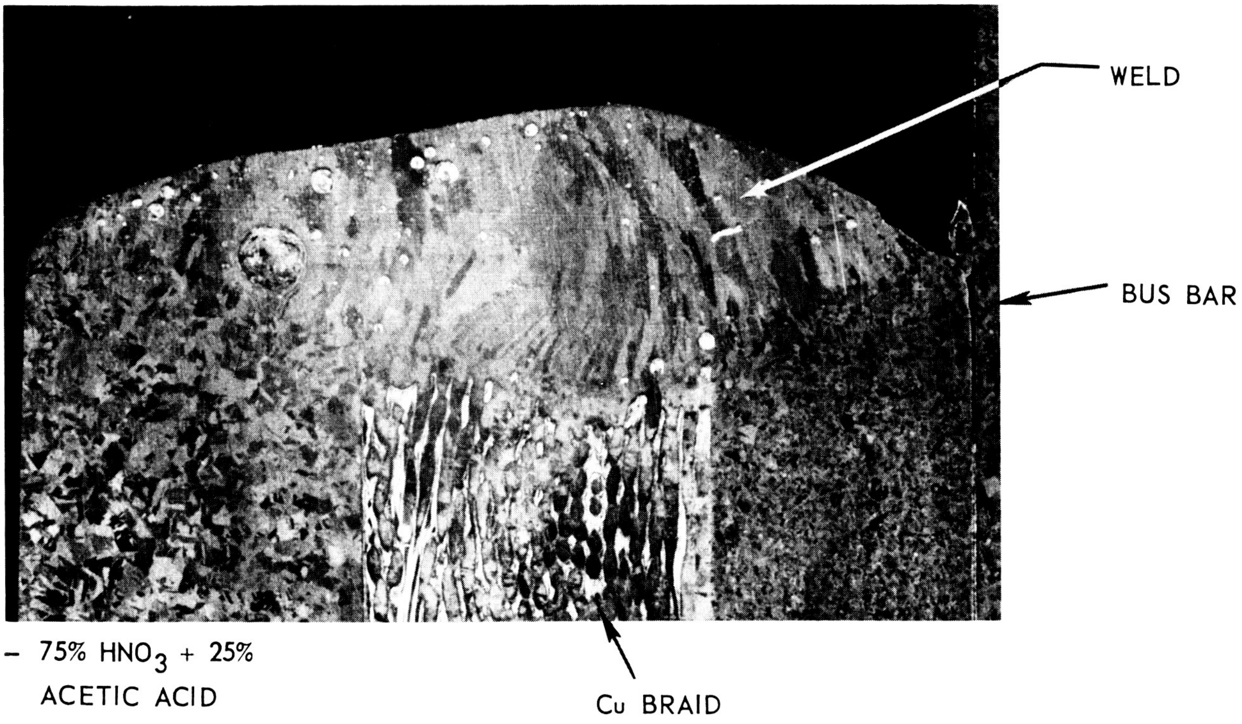
B. JOINT CROSS-SECTION

TIG-Welded Joint - Copper Braid to Terminal Strap - Configuration 1

Figure 2


WELD
BUS BAR
Cu BRAID
CLIP

L-7723 A. JOINT CONFIGURATION 2X

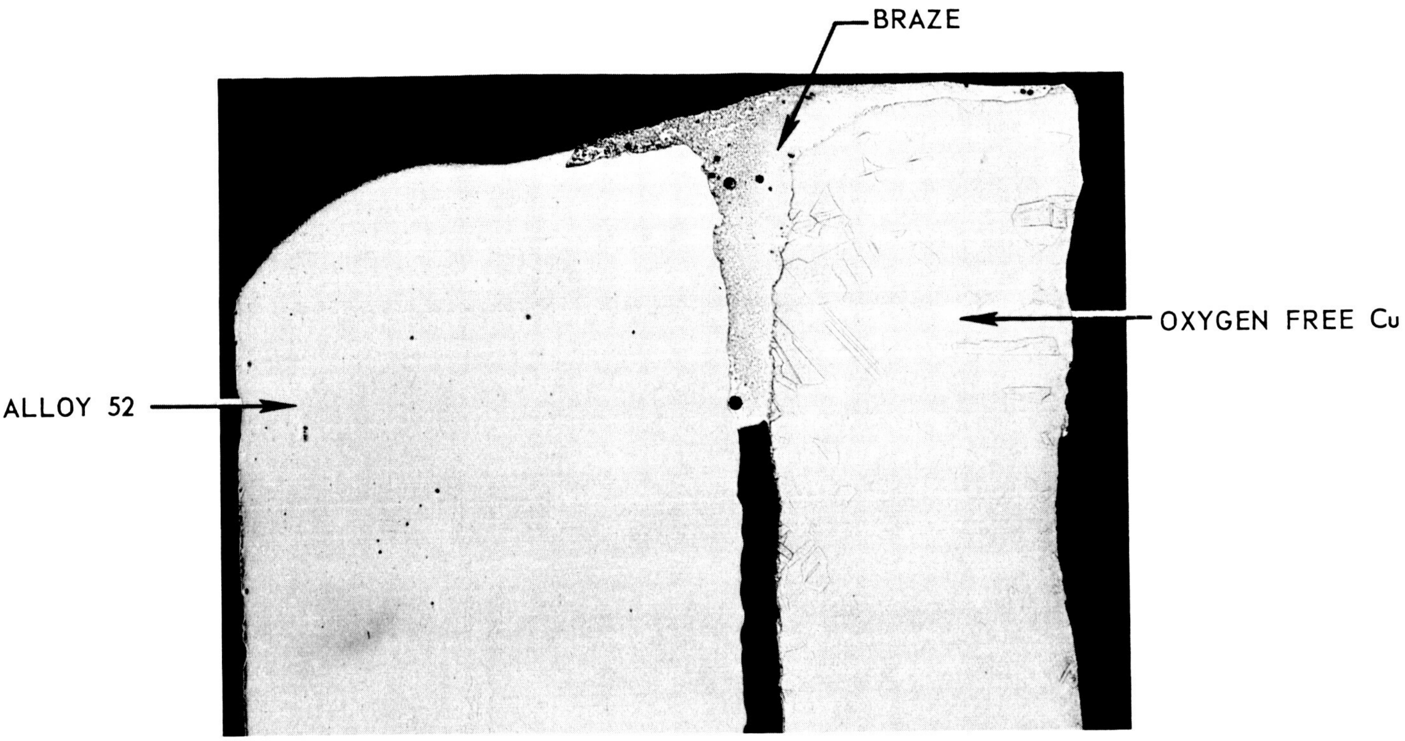

WELD
BUS BAR
Cu BRAID

ETCHANT – 75% $HNO_3$ + 25% ACETIC ACID

L-7724 B. JOINT CROSS-SECTION 15X

TIG-Welded Joint - Copper Braid
to Terminal Strap - Configuration 2

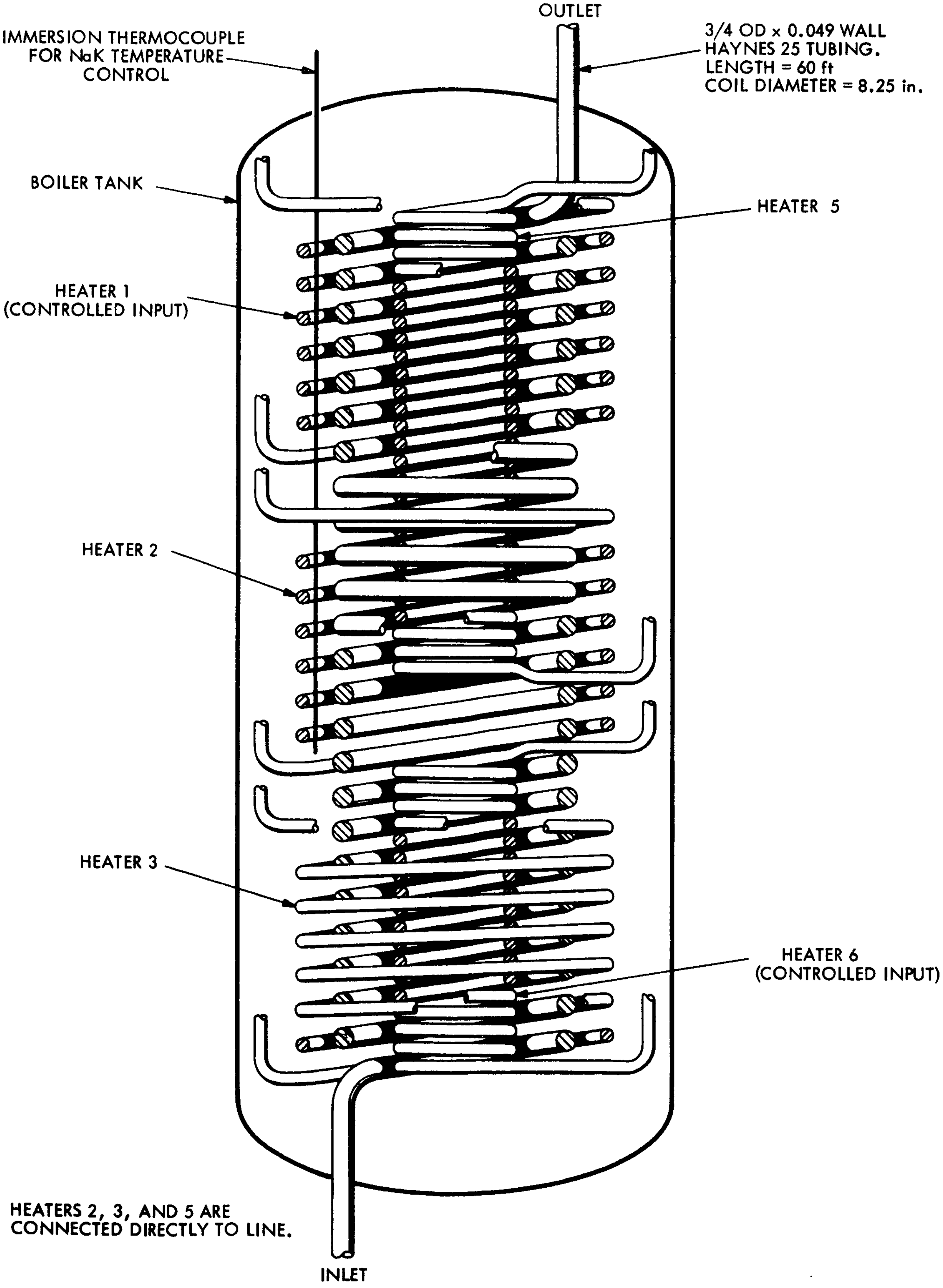
Figure 3


BRAZE
OXYGEN FREE Cu
ALLOY 52

ETCHANT - FERRIC CHLORIDE, HCl AND ALCOHOL L-7628 75 X

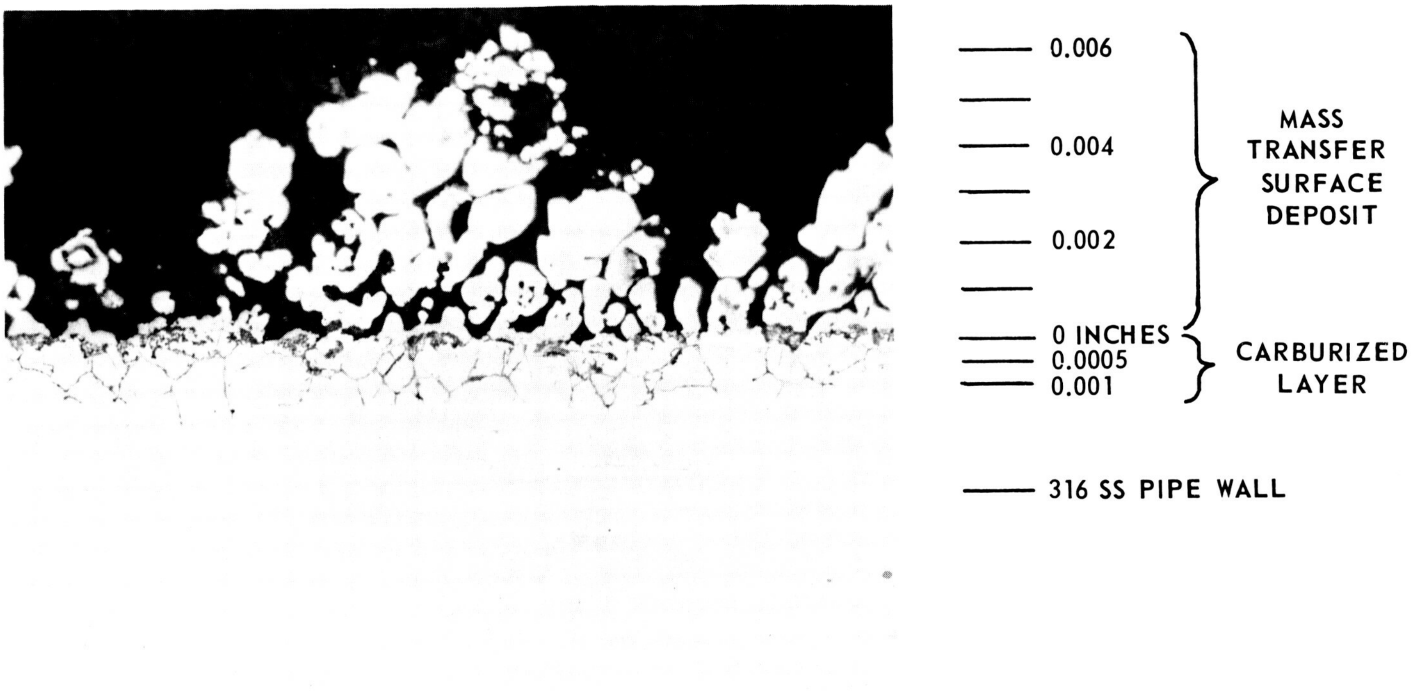
TIG-Brazed Diode Terminal Joint

Figure 4


OUTLET
IMMERSION THERMOCOUPLE FOR NaK TEMPERATURE CONTROL
3/4 OD x 0.049 WALL HAYNES 25 TUBING. LENGTH = 60 ft COIL DIAMETER = 8.25 in.
BOILER TANK
HEATER 5
HEATER 1 (CONTROLLED INPUT)
HEATER 2
HEATER 3
HEATER 6 (CONTROLLED INPUT)
HEATERS 2, 3, AND 5 ARE CONNECTED DIRECTLY TO LINE.
INLET

CTL-2 Boiler Cross Section

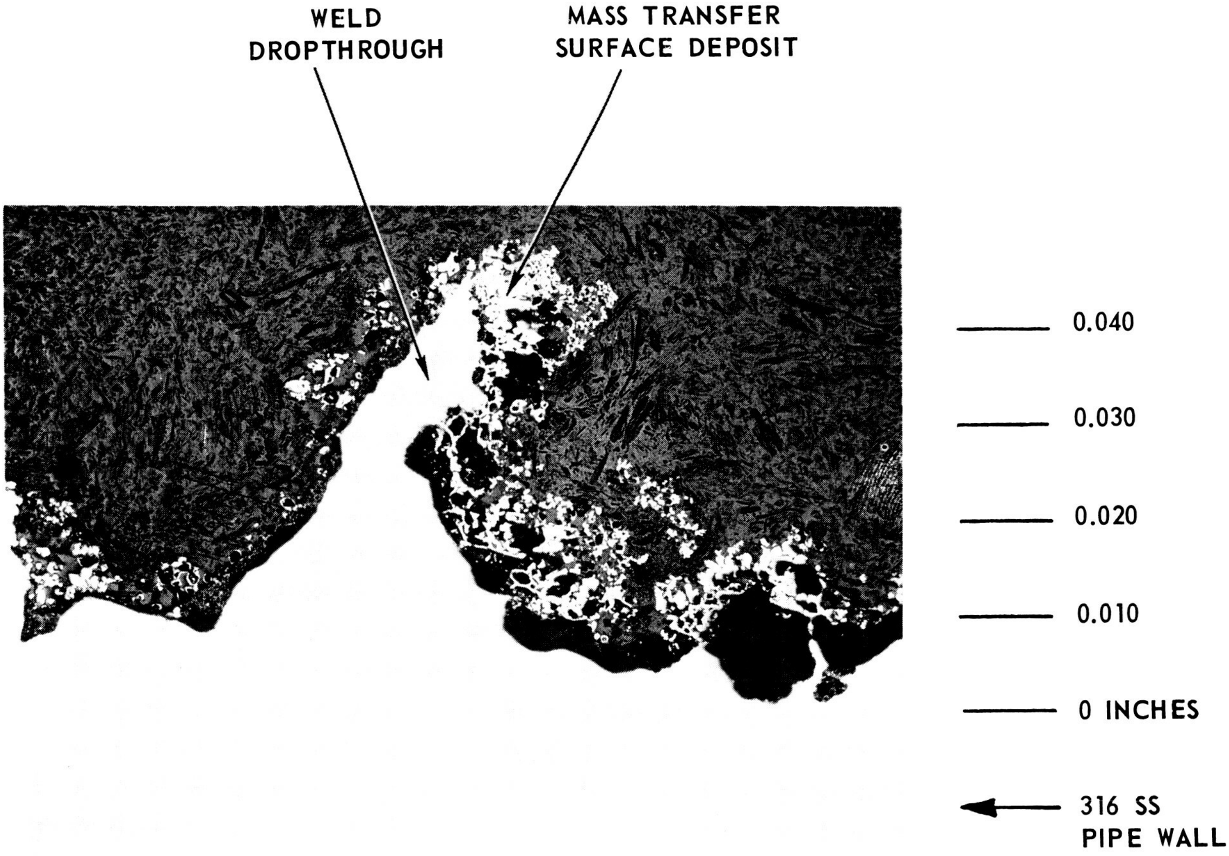
Figure 5


0.006
0.004
0.002
0 INCHES
0.0005
0.001
MASS TRANSFER SURFACE DEPOSIT
CARBURIZED LAYER
316 SS PIPE WALL

AS POLISHED L-8359 250X

Inside Surface of NaK Containment Pipe Removed from RPL-2 Primary Loop after 800 Hours Exposure Immediately Upstream of the EM Pump Inlet

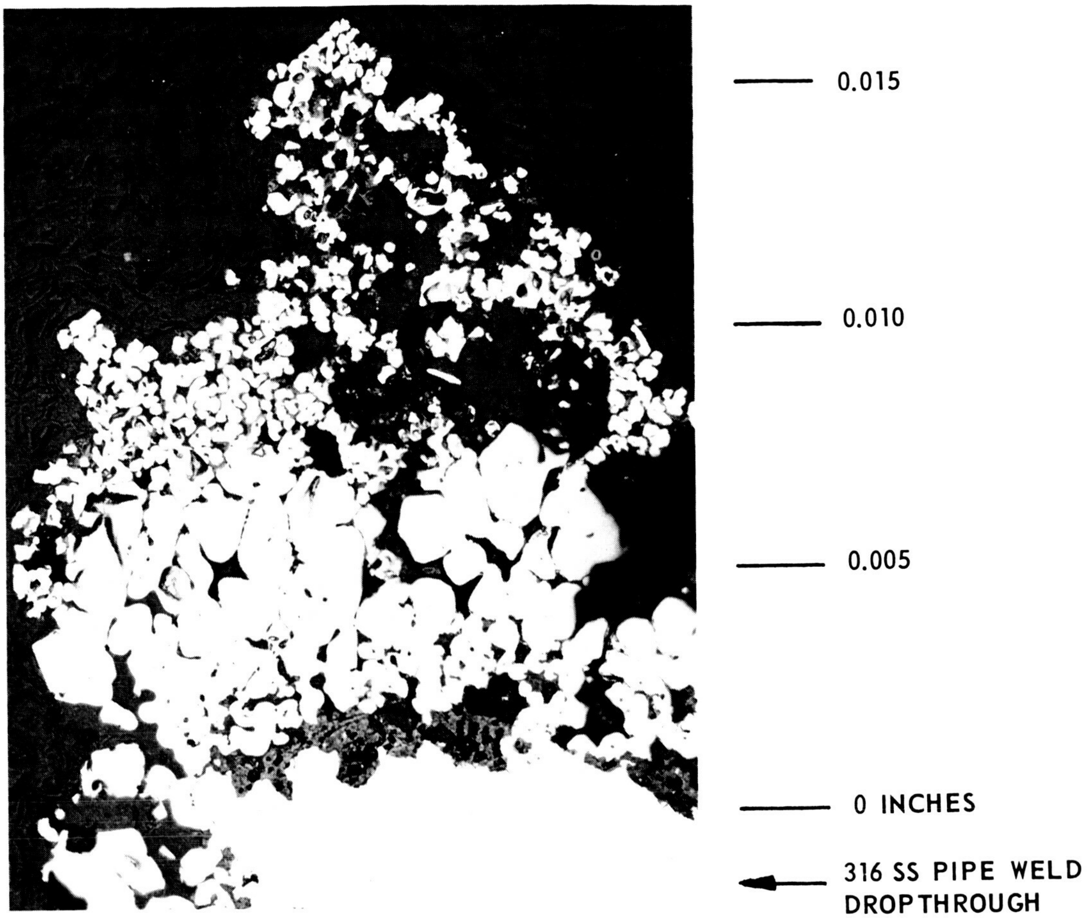
Figure 6


WELD DROPTHROUGH
MASS TRANSFER SURFACE DEPOSIT
0.040
0.030
0.020
0.010
0 INCHES
316 SS PIPE WALL

**AS POLISHED** **L-8360** **50X**

Inside Surface of NaK Containment Pipe at Butt Weld
Removed from RPL-2 Primary Loop After 800 Hours
Exposure Immediately Upstream of EM Pump Inlet

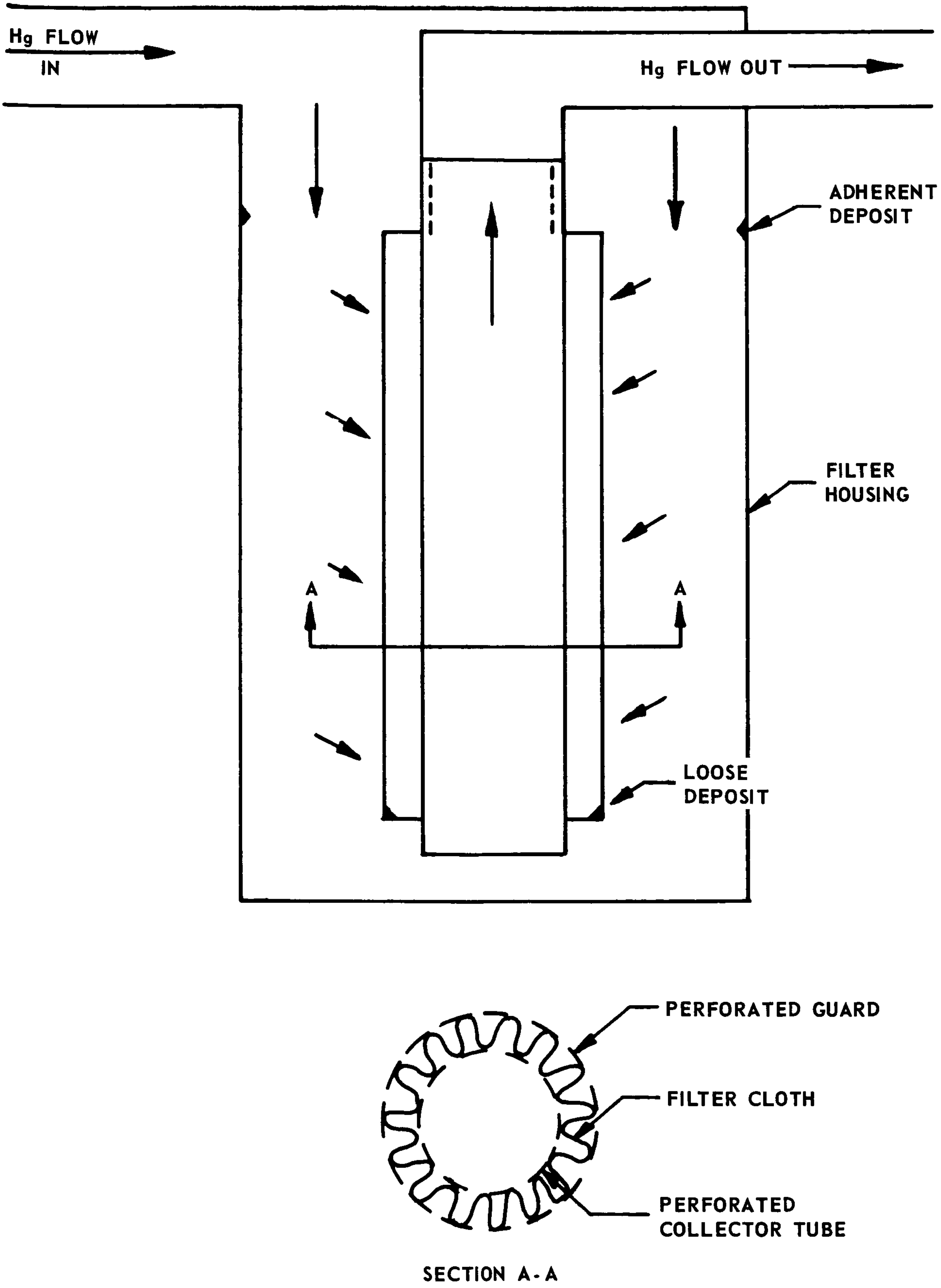
Figure 7


0.015
0.010
0.005
0 INCHES
316 SS PIPE WELD DROPTHROUGH

**AS POLISHED L-8358 250X**

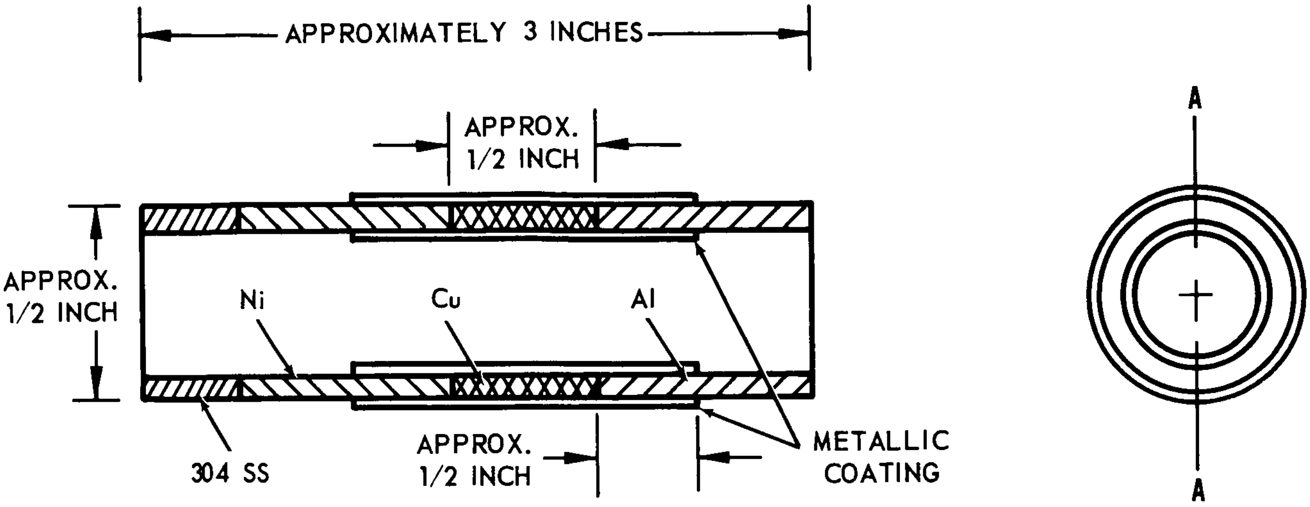
Inside Surface of NaK Containment Pipe at Butt Weld Removed from RPL-2 Primary Loop After 800 Hours Exposure Immediately Upstream of EM Pump Inlet (Expanded View of Tip of Weld Dropthrough Shown in Figure 7)

Figure 8


Hg FLOW IN
Hg FLOW OUT
ADHERENT DEPOSIT
FILTER HOUSING
A
A
LOOSE DEPOSIT
PERFORATED GUARD
FILTER CLOTH
PERFORATED COLLECTOR TUBE
SECTION A-A

Liquid Mercury Filter, RPL-2

Figure 9

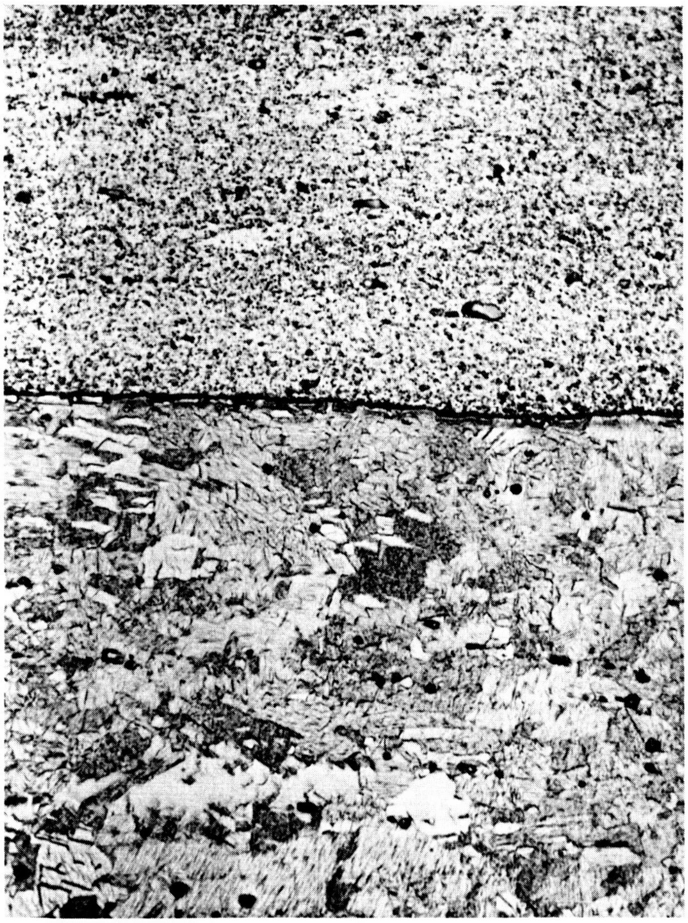

APPROXIMATELY 3 INCHES
APPROX. 1/2 INCH
APPROX. 1/2 INCH
Ni
Cu
Al
304 SS
APPROX. 1/2 INCH
METALLIC COATING
A
A

SECTION A-A
(DRAWING NOT TO SCALE)

NOTE A: METALLIC COATING TO BE ON THE OUTSIDE AND INSIDE OVER APPROXIMATE AREA SHOWN

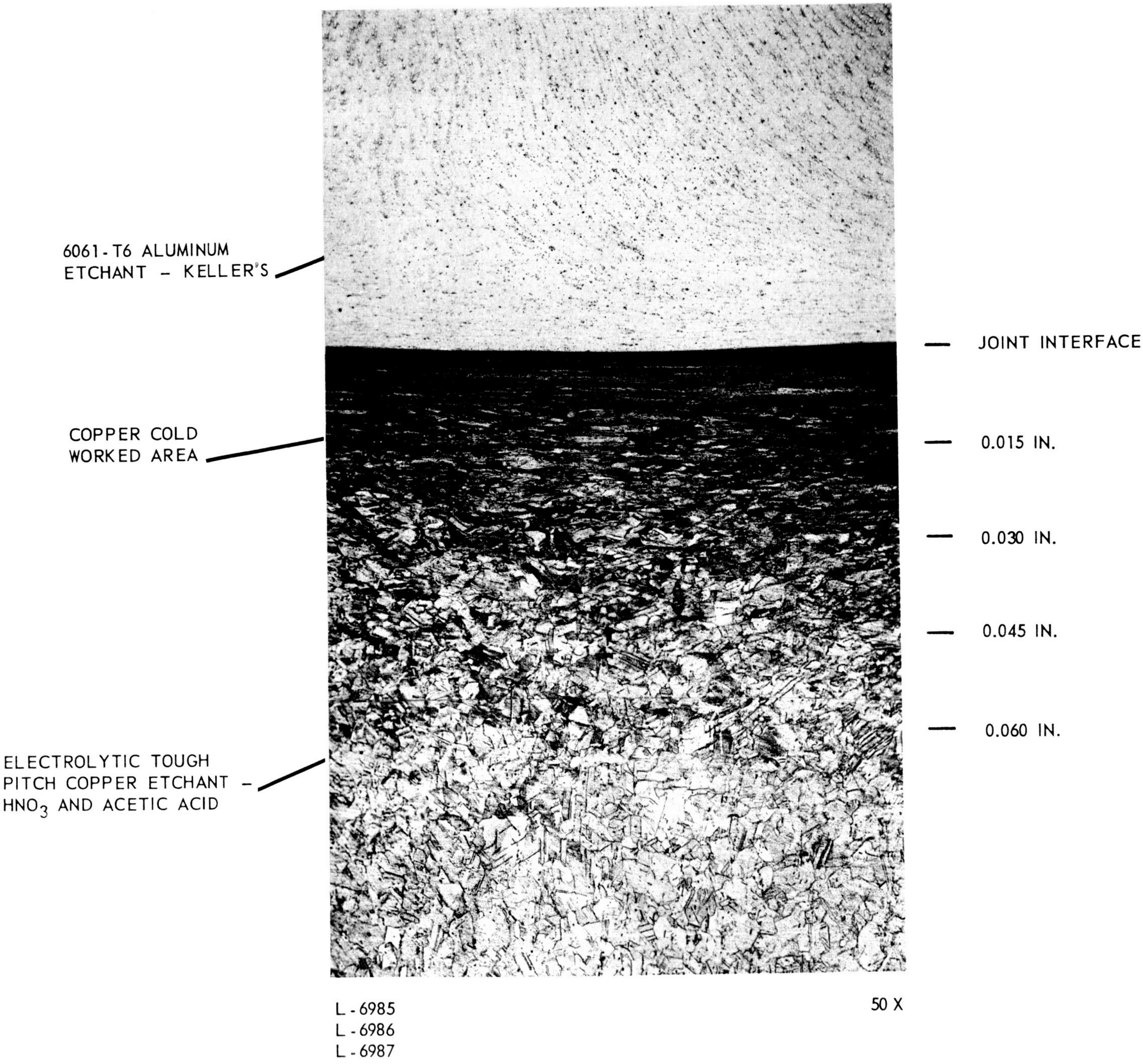
Transition Joint - Transformer-Reactor Heat Sink to Lubricant/Coolant Pump

Figure 10


ETP COPPER
ETCHANT - $HNO_3$ AND ACETIC ACID
6061 ALUMINUM ALLOY
ETCHANT - KELLER'S
L-7813
50 X

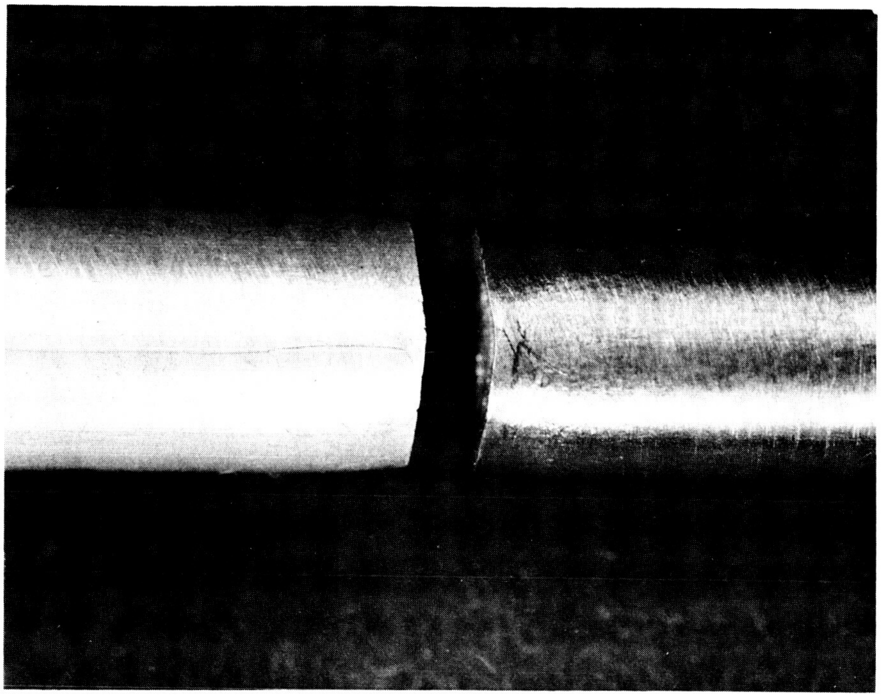
6061-T6 Aluminum/ETP Copper Interface on
Pressure Welded (Koldweld Process) Transition Joint
After 1500 Hours at 350°F

Figure 11


6061-T6 ALUMINUM
ETCHANT - KELLER'S
COPPER COLD
WORKED AREA
ELECTROLYTIC TOUGH
PITCH COPPER ETCHANT -
HNO$_3$ AND ACETIC ACID
JOINT INTERFACE
0.015 IN.
0.030 IN.
0.045 IN.
0.060 IN.
L-6985
L-6986
L-6987
50 X

6061-T6 Aluminum/ETP Copper Interface on
Pressure Welded (Koldweld Process) Transition Joint -
Produced by Kelsey-Hayes

Figure 12


6061-T6 ALUMINUM
ELECTROLYTIC TOUGH PITCH Cu
L-7939
5X

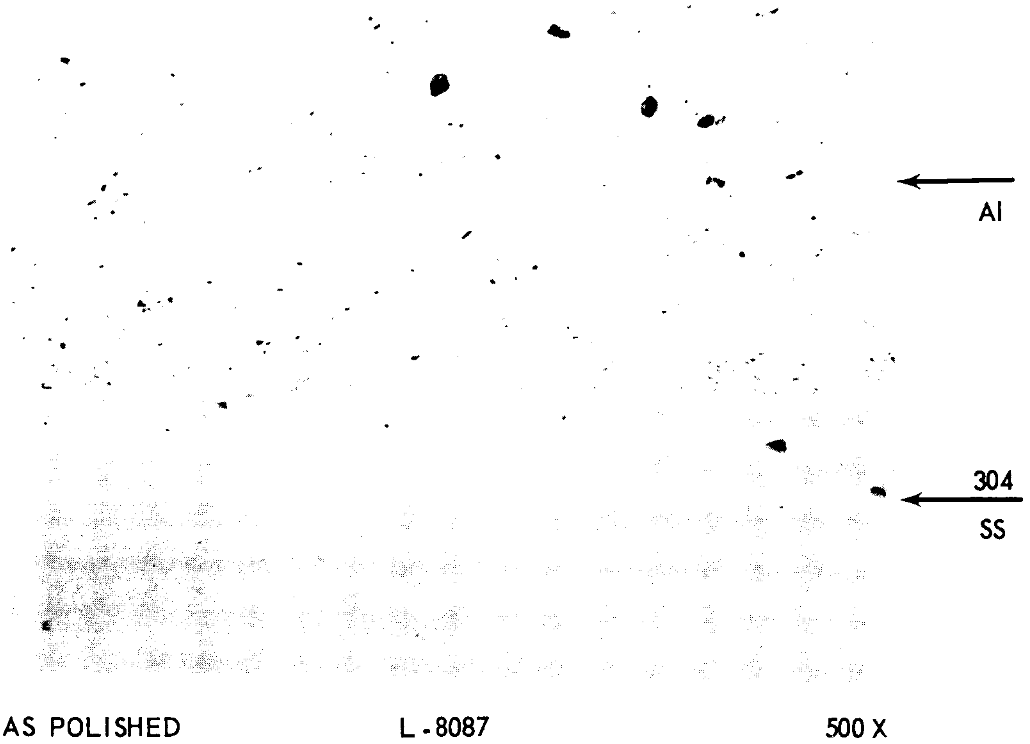
Fracture Area on Tensile Specimen of
6061-T6 Al/ETP Cu Pressure-Welded (Koldweld Process)
Transition Joint. Specimen Tested at 75°F
After 1500 Hours at 350°F

Figure 13

Al
304 SS
AS POLISHED
L-7726
500 X

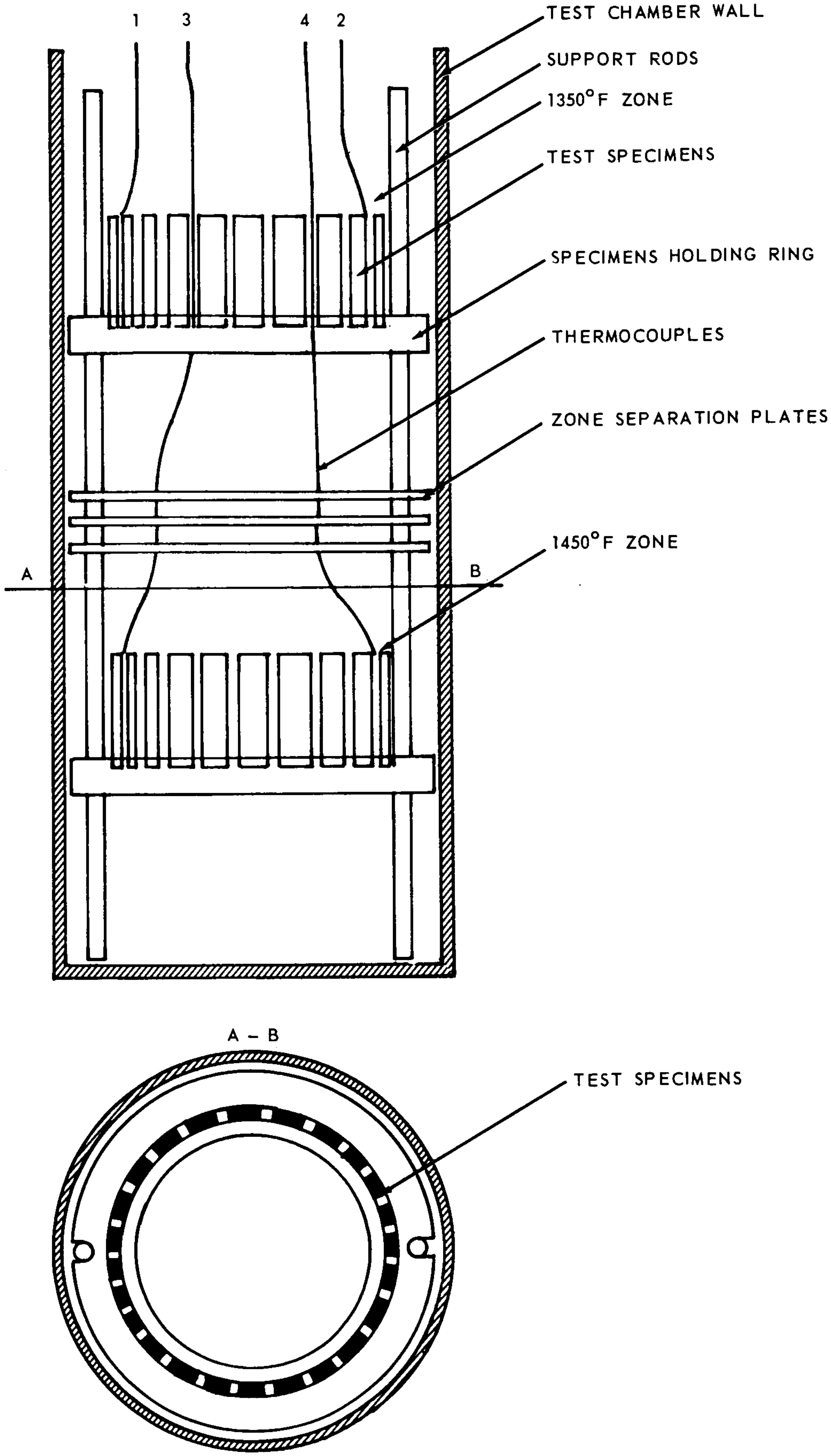
Al/304 SS Interface on Coextruded Tubular
Transition Joint - Produced by Nuclear Metals, Inc.

Figure 14


Al
304
SS
AS POLISHED
L-8087
500 X

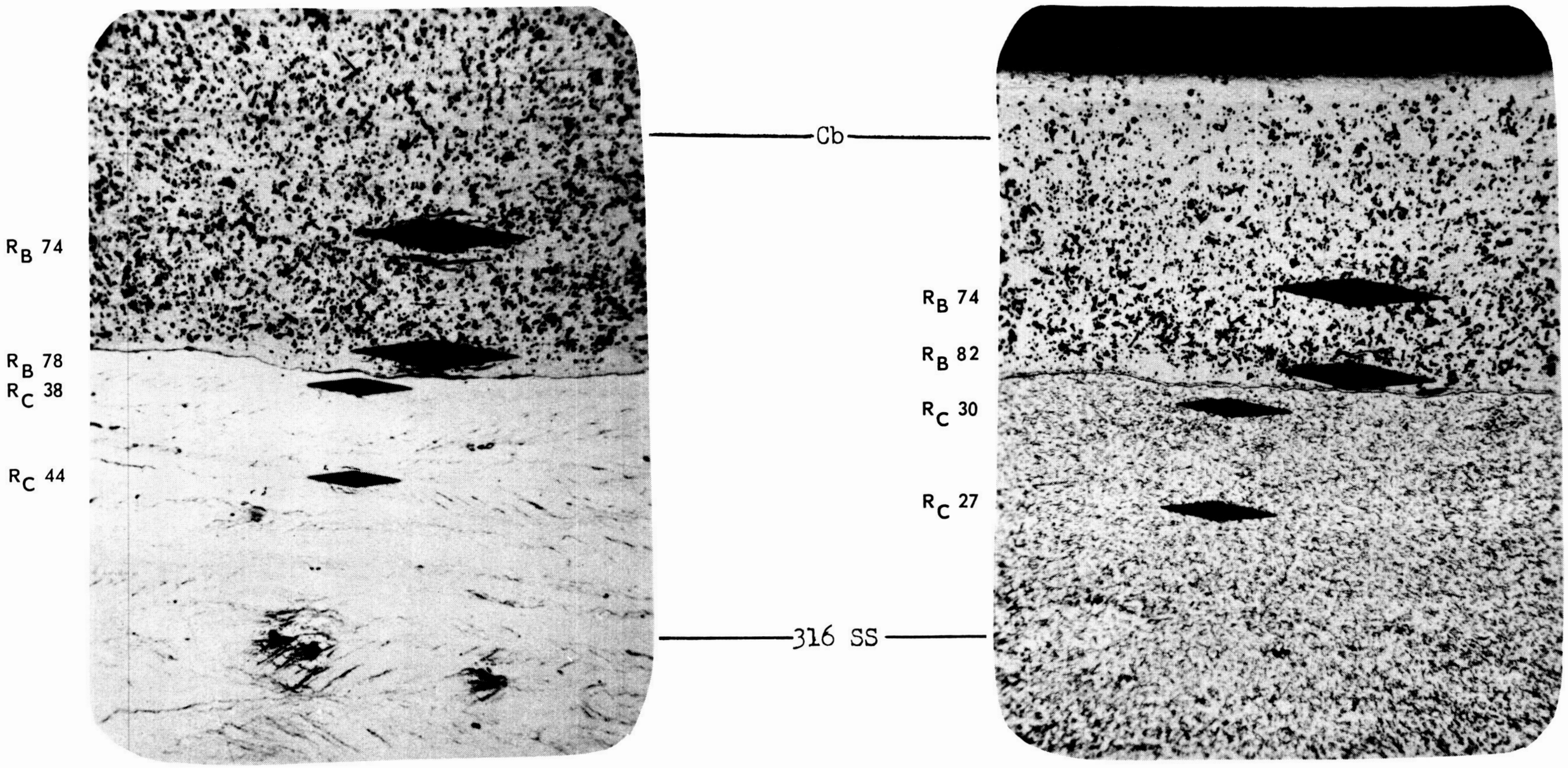
Al/304 SS Interface on Coextruded Tubular
Transition Joint After 350 Hours at 275°F

Figure 15


1
3
4
2
TEST CHAMBER WALL
SUPPORT RODS
1350°F ZONE
TEST SPECIMENS
SPECIMENS HOLDING RING
THERMOCOUPLES
ZONE SEPARATION PLATES
1450°F ZONE
A
B
A – B
TEST SPECIMENS

Test Specimens Holder for Thermal Exposure Study

Figure 16


$R_B$ 74
$R_B$ 78
$R_C$ 38
$R_C$ 44
Cb
316 SS
$R_B$ 74
$R_B$ 82
$R_C$ 30
$R_C$ 27

Etchant: 10 cc $H_2O$ LRC 1-1 275X
+5 cc $HNO_3$
+2 cc HF

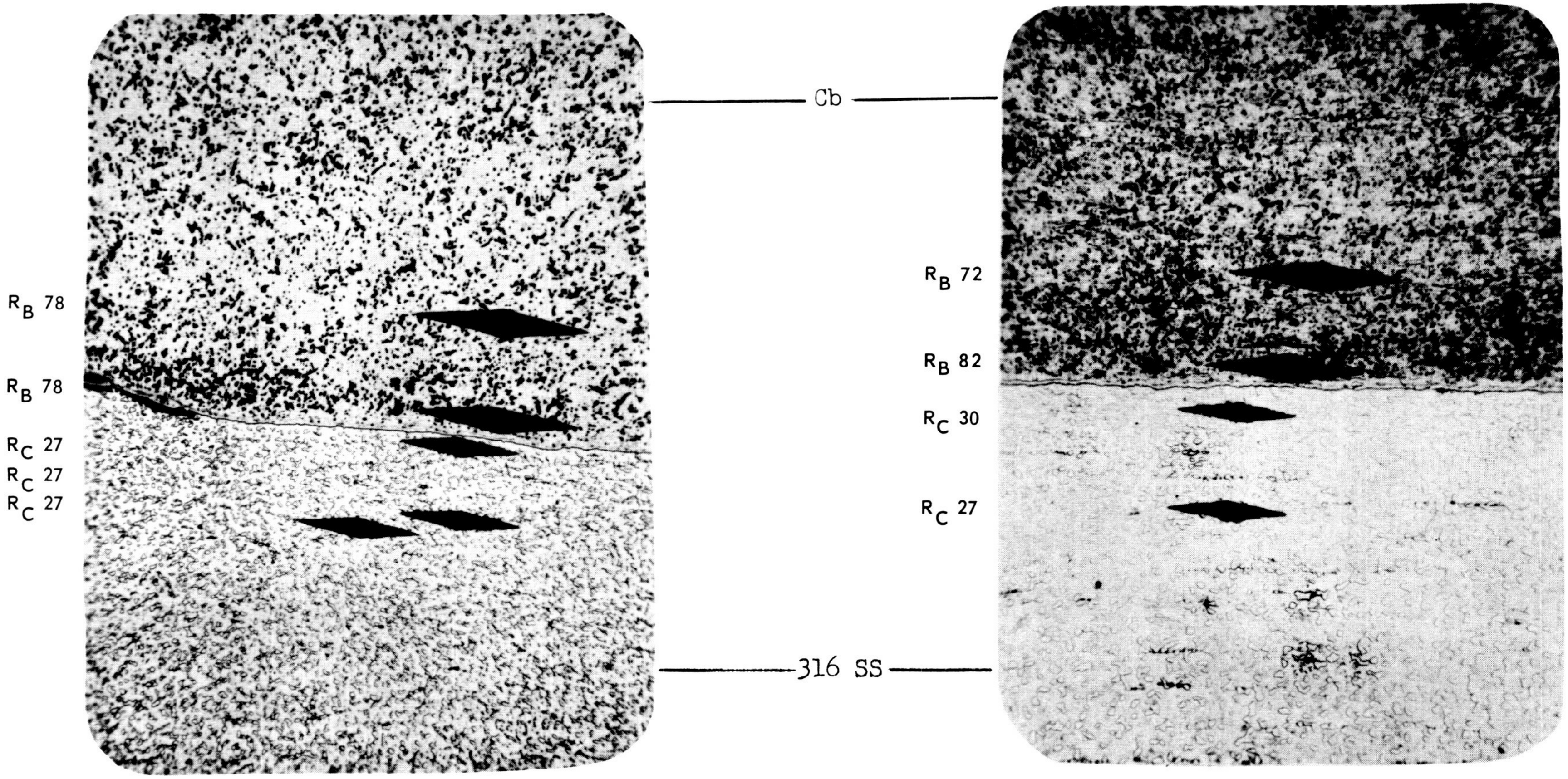
Etchant: 10 cc $H_2O$ LRC 1-4 275X
+5 cc $HNO_3$
+2 cc HF

a. Pre-Exposure Specimen

b. After 2500 Hours at 1350°F in Vacuum ($10^{-5}$torr)

High-Temperature Diffusion of Cb/316 SS Flat Sheet Specimen

Figure 17



$R_B$ 78
$R_B$ 78
$R_C$ 27
$R_C$ 27
$R_C$ 27
Cb
316 SS
$R_B$ 72
$R_B$ 82
$R_C$ 30
$R_C$ 27

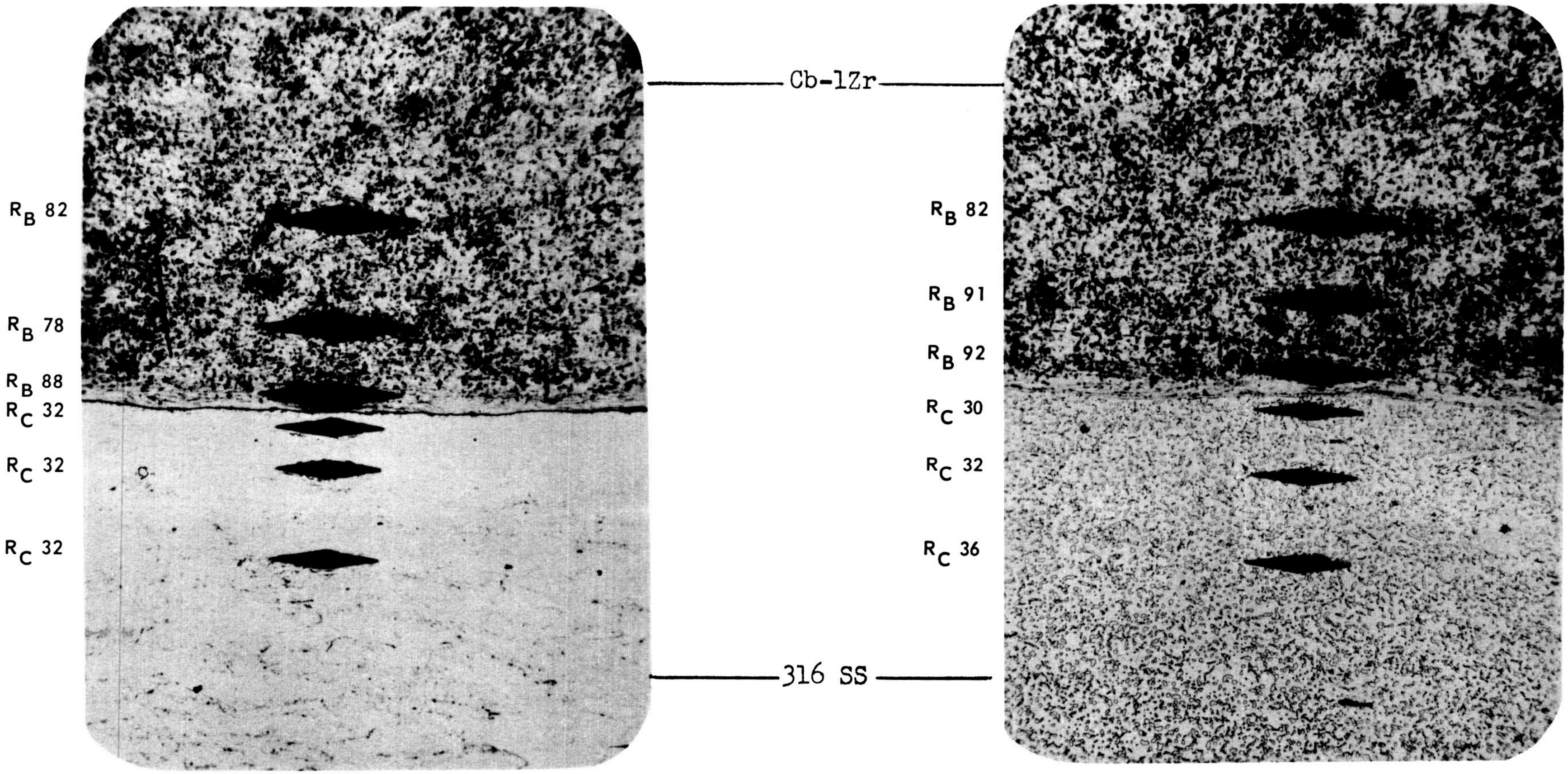
Etchant: 10 cc $H_2O$ +5 cc $HNO_3$ +2 cc HF LRC 1-7 275X

Etchant: 10 cc $H_2O$ +5 cc $HNO_3$ +2 cc HF LRC 2-7 275X

a. Flat Sheet

b. Tube

Diffusion of Cb/316 SS Flat Sheet and Tube Specimens
After 2500 Hours at 1450°F in Vacuum ($10^{-5}$torr)


Cb-1Zr
316 SS
$R_B$ 82
$R_B$ 78
$R_B$ 88
$R_C$ 32
$R_C$ 32
$R_C$ 32
$R_B$ 82
$R_B$ 91
$R_B$ 92
$R_C$ 30
$R_C$ 32
$R_C$ 36

Etchant: 10 cc $H_2O$ LRC 7-1 275X
+5 cc $HNO_3$
+2 cc HF

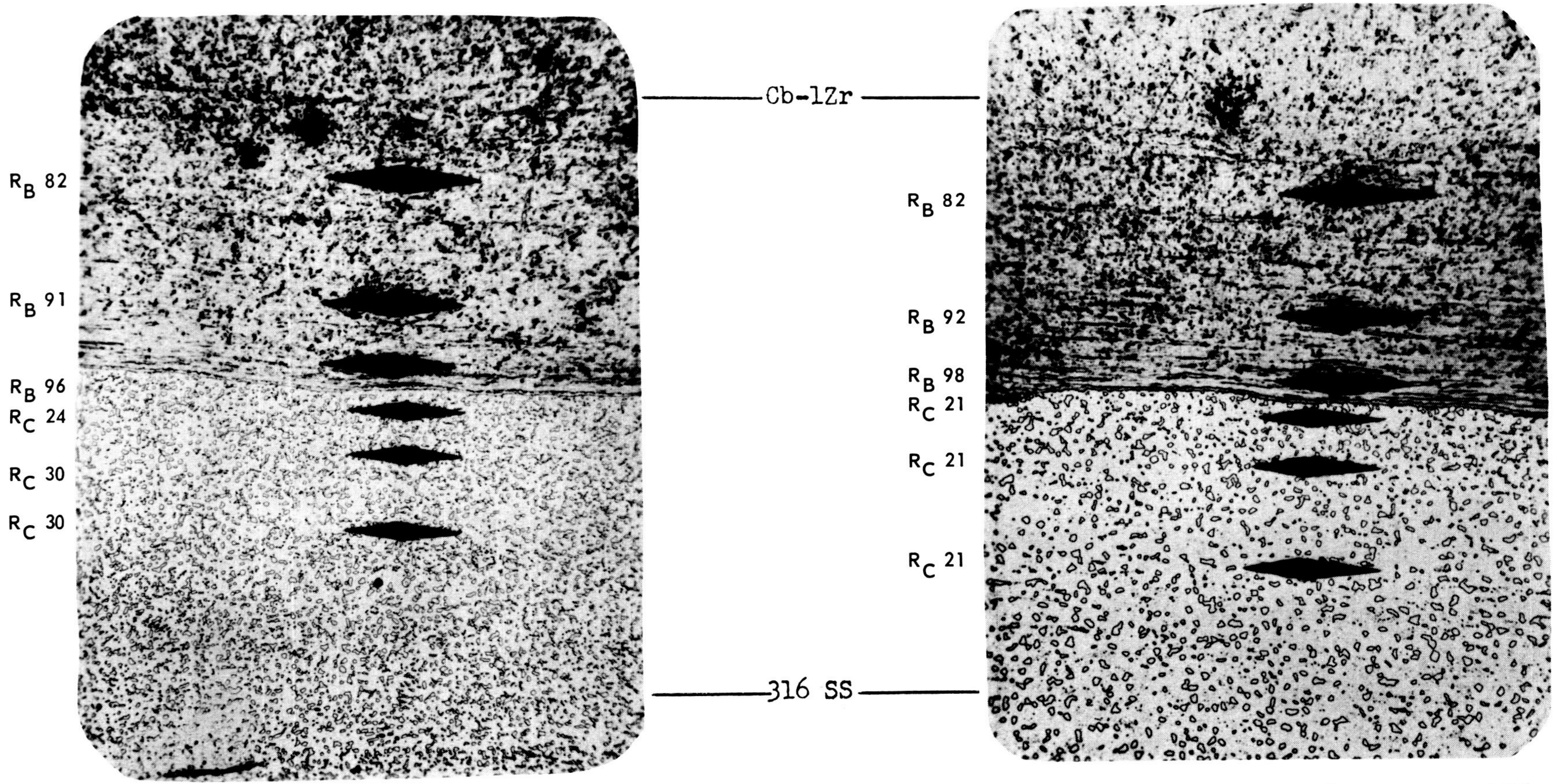
**a.** Pre-Exposure Specimen

Etchant: 10 cc $H_2O$ LRC 7-4 275X
+5 cc $HNO_3$
+2 cc HF

b. After 2500 Hours at 1350°F
in Vacuum ($10^{-5}$torr)

High-Temperature Diffusion of Cb-1Zr/316 SS Flat Sheet Specimen

Figure 19


Cb-1Zr
316 SS
$R_B$ 82
$R_B$ 91
$R_B$ 96
$R_C$ 24
$R_C$ 30
$R_C$ 30
$R_B$ 82
$R_B$ 92
$R_B$ 98
$R_C$ 21
$R_C$ 21
$R_C$ 21

Etchant: 10 cc $H_2O$ LRC 7-7 275X
+5 cc $HNO_3$
+2 cc HF

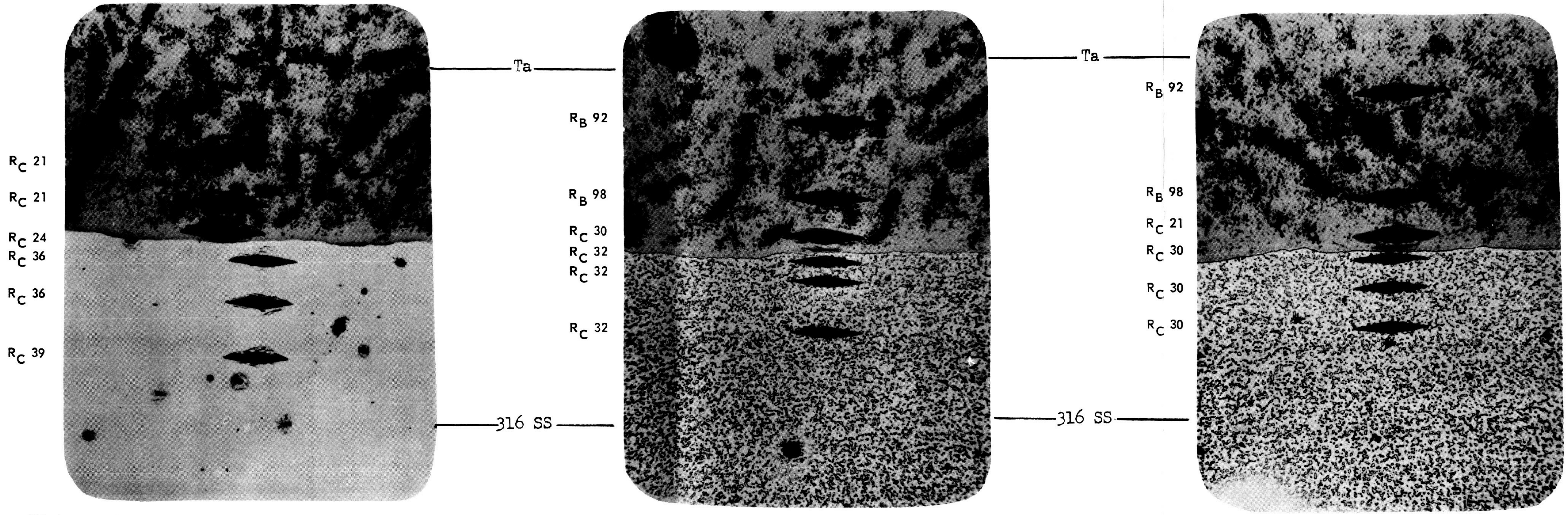
Etchant: 10 cc $H_2O$ LRC 8-7 275X
+5 cc $HNO_3$
+2 cc HF

a. Flat Sheet

b. Tube

Diffusion of Cb-1Zr/316 SS Flat Sheet and Tube Specimens
After 2500 Hours at 1450°F in Vacuum ($10^{-5}$torr)

Figure 20


$R_C$ 21
$R_C$ 21
$R_C$ 24
$R_C$ 36
$R_C$ 36
$R_C$ 39
Ta
316 SS
$R_B$ 92
$R_B$ 98
$R_C$ 30
$R_C$ 32
$R_C$ 32
$R_C$ 32
Ta
316 SS
$R_B$ 92
$R_B$ 98
$R_C$ 21
$R_C$ 30
$R_C$ 30
$R_C$ 30

Etchant: 10 cc $H_2O$ +5 cc $HNO_3$ +2 cc HF LRC 13-1 275X

a. Pre-Exposure Specimen

Etchant: 10 cc $H_2O$ +5 cc $HNO_3$ +2 cc HF LRC 13-4 275X

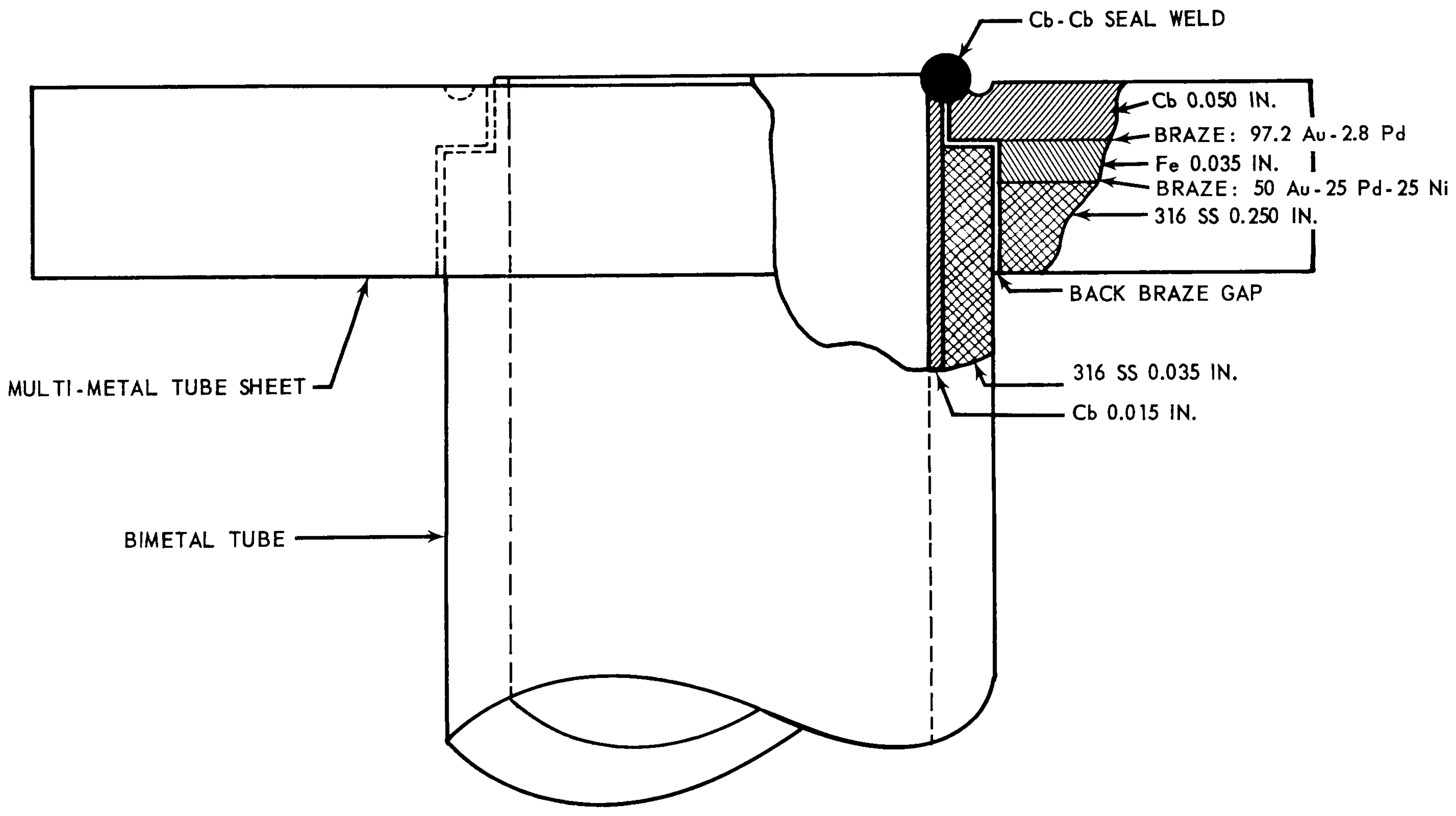
b. After 2500 Hours at 1350°F in Vacuum ($10^{-5}$torr)

Etchant: 10 cc $H_2O$ +5 cc $HNO_3$ +2 cc HF LRC 13-7 275X

c. After 2500 Hours at 1450°F in Vacuum ($10^{-5}$torr)

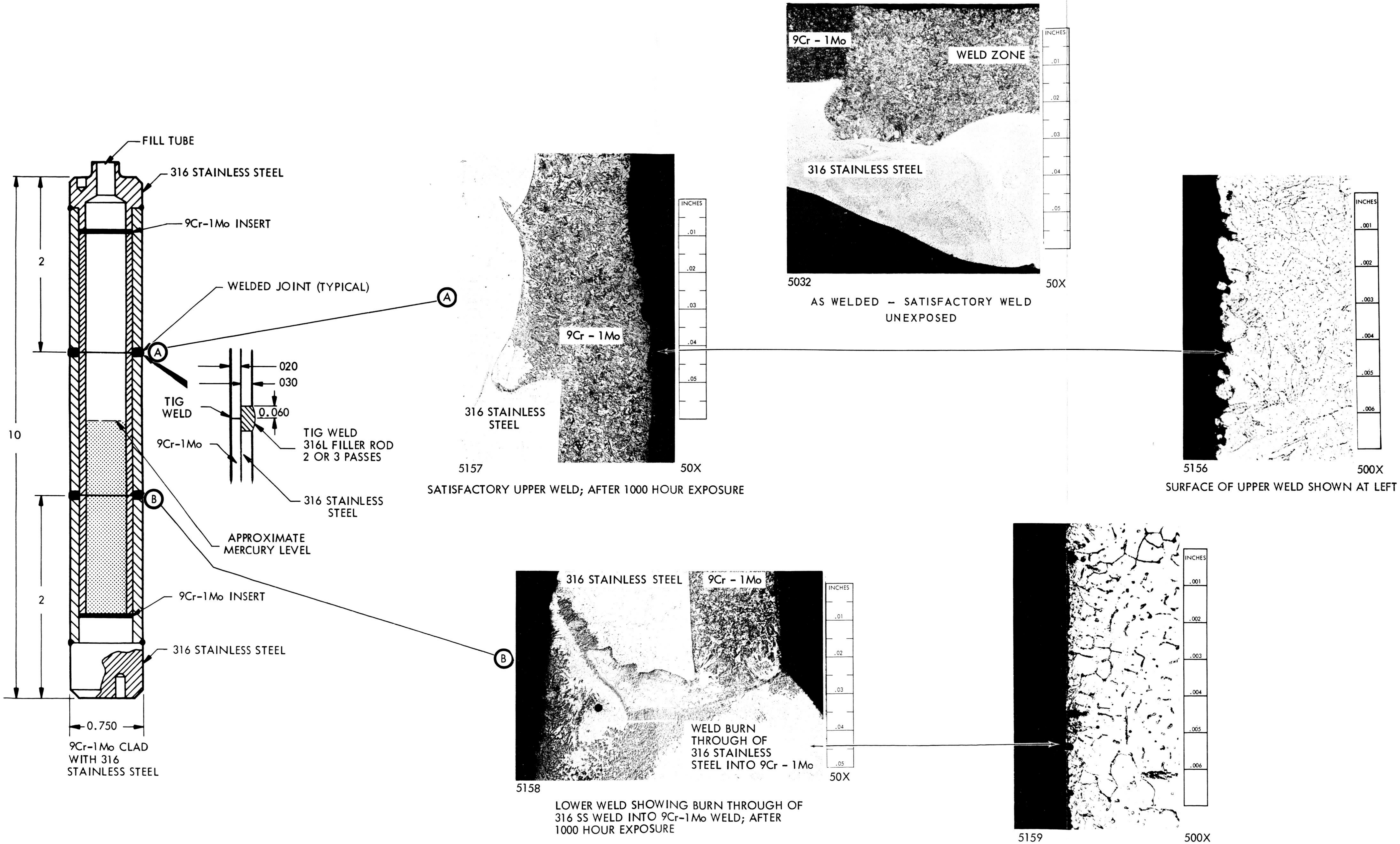
High-Temperature Diffusion of Ta/316 SS Flat Sheet Specimens

Figure 21


Cb-Cb SEAL WELD
Cb 0.050 IN.
BRAZE: 97.2 Au-2.8 Pd
Fe 0.035 IN.
BRAZE: 50 Au-25 Pd-25 Ni
316 SS 0.250 IN.
BACK BRAZE GAP
316 SS 0.035 IN.
Cb 0.015 IN.
MULTI-METAL TUBE SHEET
BIMETAL TUBE

Refractory Mercury Containment Tube-to-Tube Sheet Back-Brazed Joint

Figure 22


FILL TUBE
316 STAINLESS STEEL
9Cr-1Mo INSERT
WELDED JOINT (TYPICAL)
A
B
2
10
2
TIG WELD
9Cr-1Mo
020
030
0.060
TIG WELD 316L FILLER ROD 2 OR 3 PASSES
316 STAINLESS STEEL
APPROXIMATE MERCURY LEVEL
9Cr-1Mo INSERT
316 STAINLESS STEEL
0.750
9Cr-1Mo CLAD WITH 316 STAINLESS STEEL
9Cr - 1Mo
316 STAINLESS STEEL
INCHES
.01
.02
.03
.04
.05
WELD ZONE
9Cr - 1Mo
316 STAINLESS STEEL
.001
.002
.003
.004
.005
.006
316 STAINLESS STEEL
9Cr - 1Mo
WELD BURN THROUGH OF 316 STAINLESS STEEL INTO 9Cr - 1Mo

5157 50X
SATISFACTORY UPPER WELD; AFTER 1000 HOUR EXPOSURE

5032 50X
AS WELDED – SATISFACTORY WELD UNEXPOSED

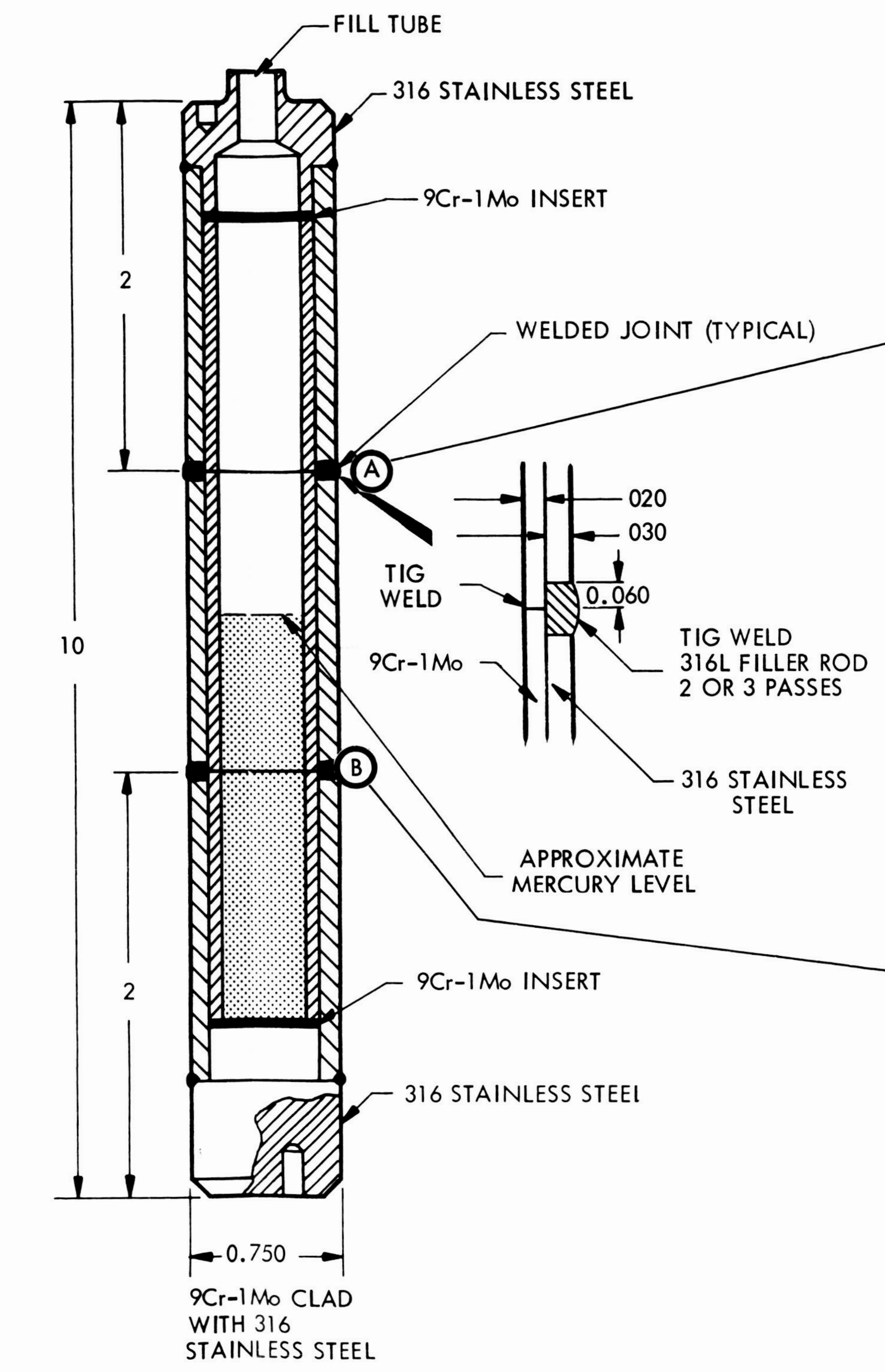
5156 500X
SURFACE OF UPPER WELD SHOWN AT LEFT

5158 50X
LOWER WELD SHOWING BURN THROUGH OF 316 SS WELD INTO 9Cr-1Mo WELD; AFTER 1000 HOUR EXPOSURE

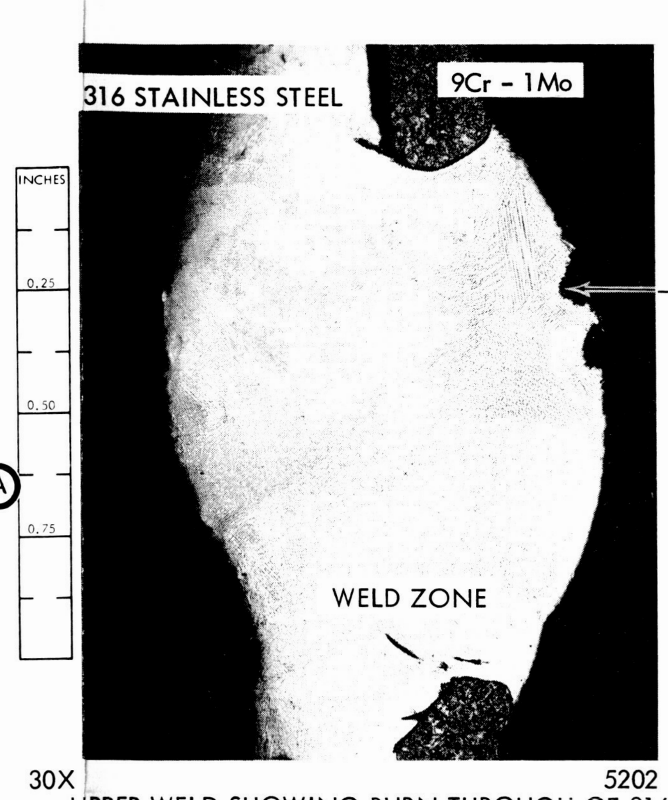
5159 500X
SURFACE OF LOWER WELD SHOWN AT LEFT

Reflux Capsule, 9Cr-1Mo Clad with 316 SS (Test Temperature 1150°F)

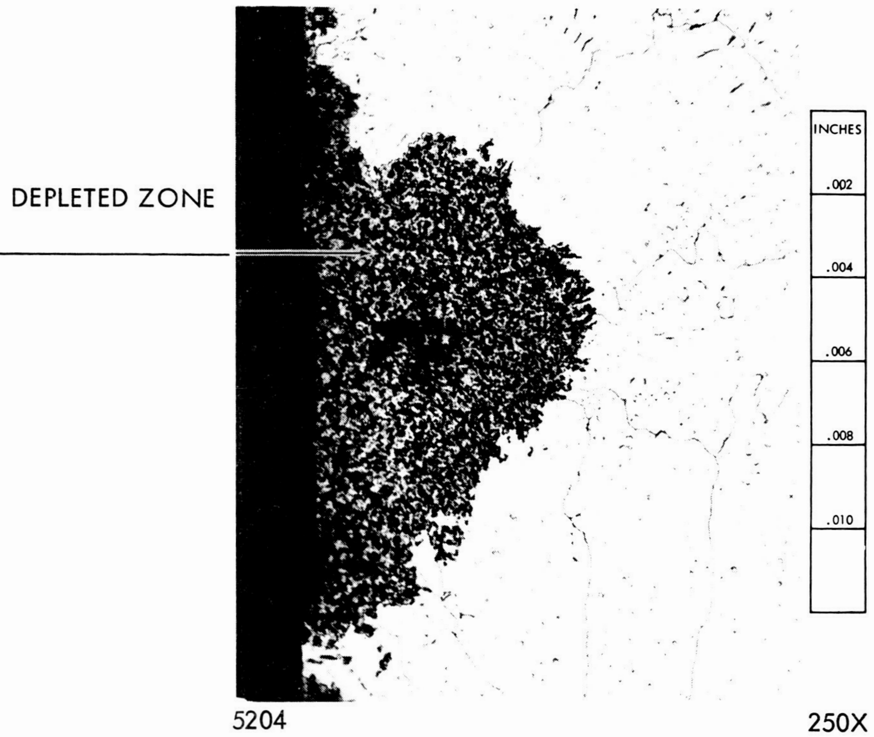
Figure 23


FILL TUBE
316 STAINLESS STEEL
9Cr-1Mo INSERT
WELDED JOINT (TYPICAL)
A
020
030
0.060
TIG WELD
9Cr-1Mo
TIG WELD
316L FILLER ROD
2 OR 3 PASSES
316 STAINLESS STEEL
B
APPROXIMATE MERCURY LEVEL
9Cr-1Mo INSERT
316 STAINLESS STEEL
2
10
2
0.750
9Cr-1Mo CLAD
WITH 316
STAINLESS STEEL

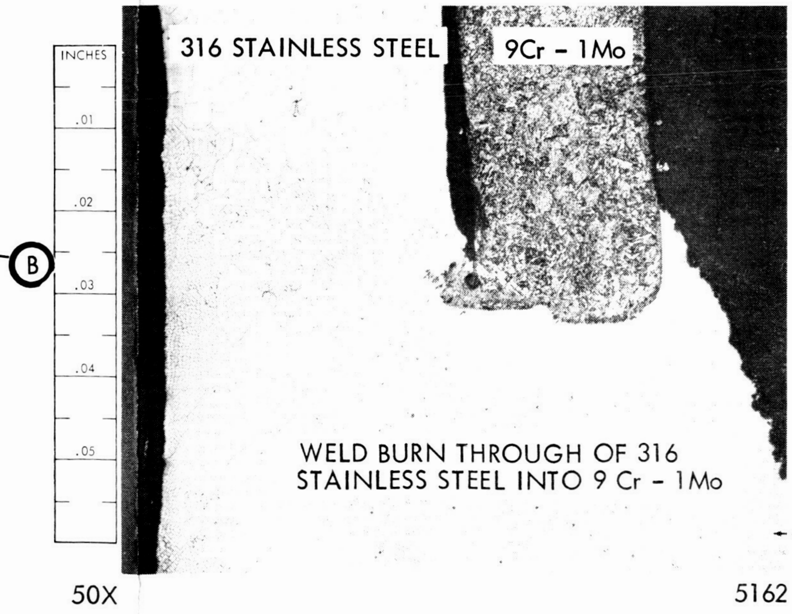
NOTE: DIMENSIONS IN INCHES


A
316 STAINLESS STEEL
9Cr - 1Mo
WELD ZONE
INCHES
0.25
0.50
0.75
30X
5202

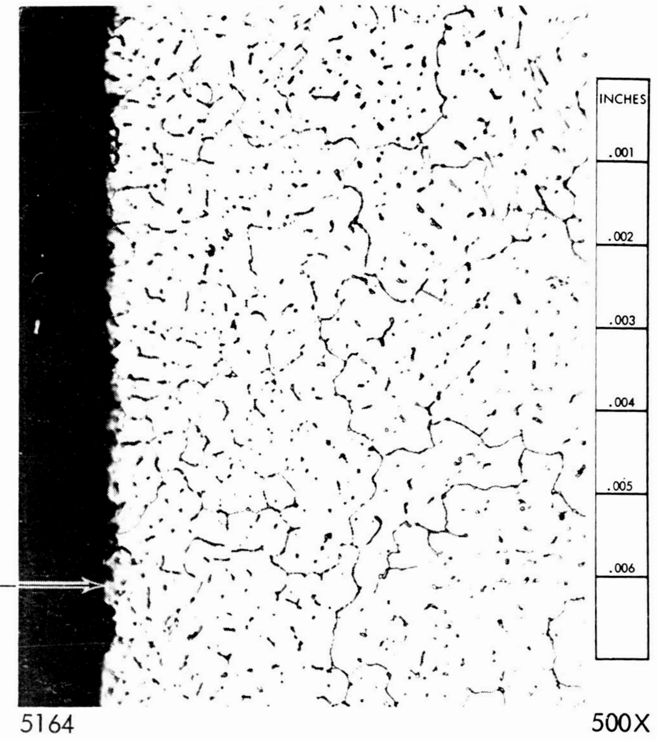
UPPER WELD SHOWING BURN THROUGH OF 316 STAINLESS STEEL WELD INTO 9Cr - 1Mo WELD; AFTER 1000 HOUR EXPOSURE. NOTE DEPLETED ZONE INDICATING MERCURY CORROSION


DEPLETED ZONE
INCHES
.002
.004
.006
.008
.010
5204
250X

SURFACE OF UPPER WELD SHOWN AT LEFT


B
316 STAINLESS STEEL
9Cr - 1Mo
WELD BURN THROUGH OF 316
STAINLESS STEEL INTO 9 Cr - 1Mo
INCHES
.01
.02
.03
.04
.05
50X
5162

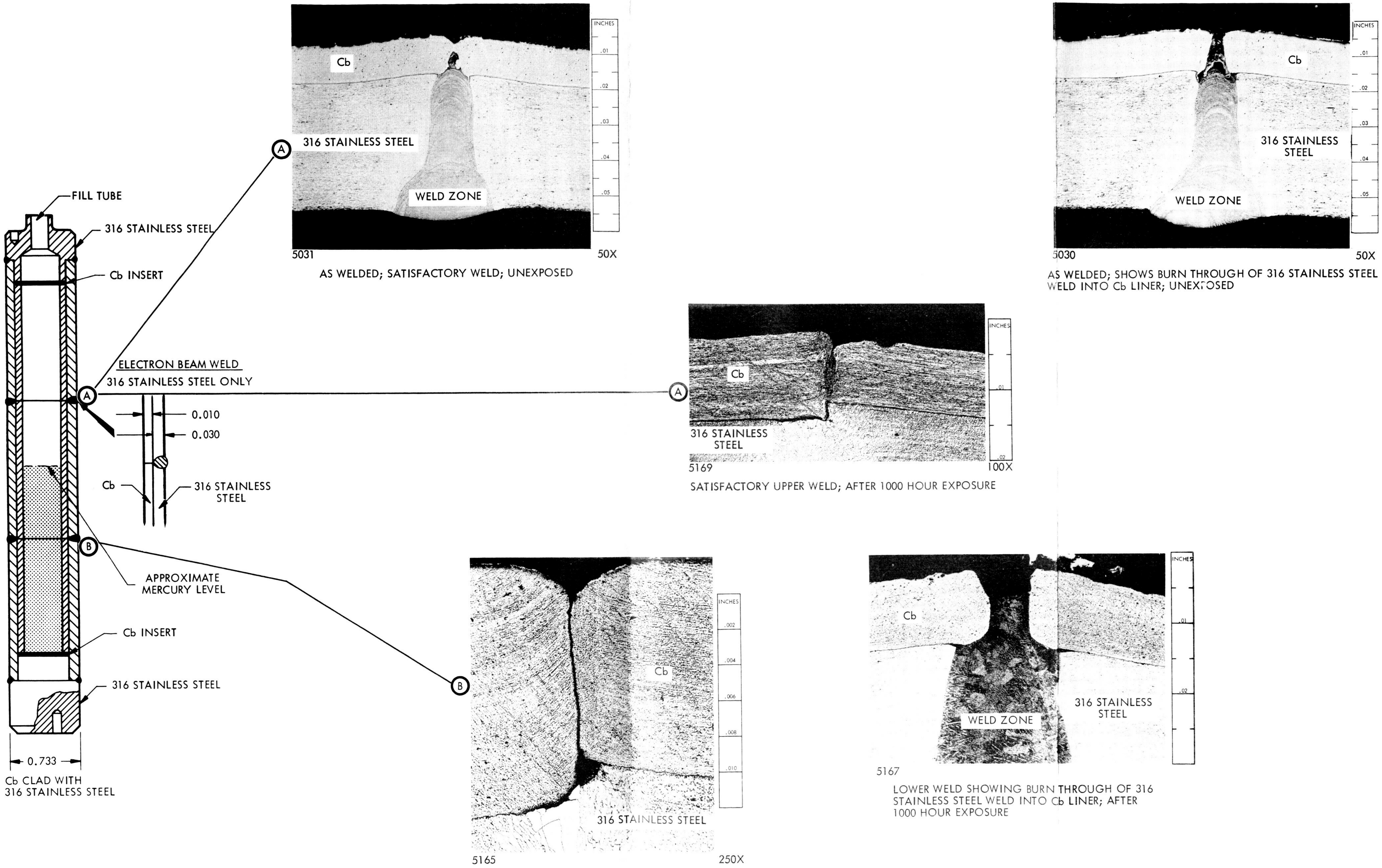
LOWER WELD SHOWING BURN THROUGH OF 316 STAINLESS STEEL WELD INTO 9Cr - 1Mo WELD; AFTER 1000 HOUR EXPOSURE


INCHES
.001
.002
.003
.004
.005
.006
5164
500X

SURFACE OF LOWER WELD SHOWN AT LEFT

Reflux Capsule, 9Cr-1Mo Clad with 316 SS (Test Temperature 1150°F)

Figure 24


FILL TUBE
316 STAINLESS STEEL
Cb INSERT
ELECTRON BEAM WELD
316 STAINLESS STEEL ONLY
A
0.010
0.030
Cb
316 STAINLESS STEEL
B
APPROXIMATE MERCURY LEVEL
Cb INSERT
316 STAINLESS STEEL
0.733
Cb CLAD WITH 316 STAINLESS STEEL
A
Cb
316 STAINLESS STEEL
WELD ZONE
INCHES
.01
.02
.03
.04
.05
5031
50X
Cb
316 STAINLESS STEEL
WELD ZONE
INCHES
.01
.02
.03
.04
.05
5030
50X
A
Cb
316 STAINLESS STEEL
INCHES
.01
.02
5169
100X
B
Cb
316 STAINLESS STEEL
INCHES
.002
.004
.006
.008
.010
5165
250X
Cb
316 STAINLESS STEEL
WELD ZONE
INCHES
.01
.02
5167

AS WELDED; SATISFACTORY WELD; UNEXPOSED

AS WELDED; SHOWS BURN THROUGH OF 316 STAINLESS STEEL WELD INTO Cb LINER; UNEXPOSED

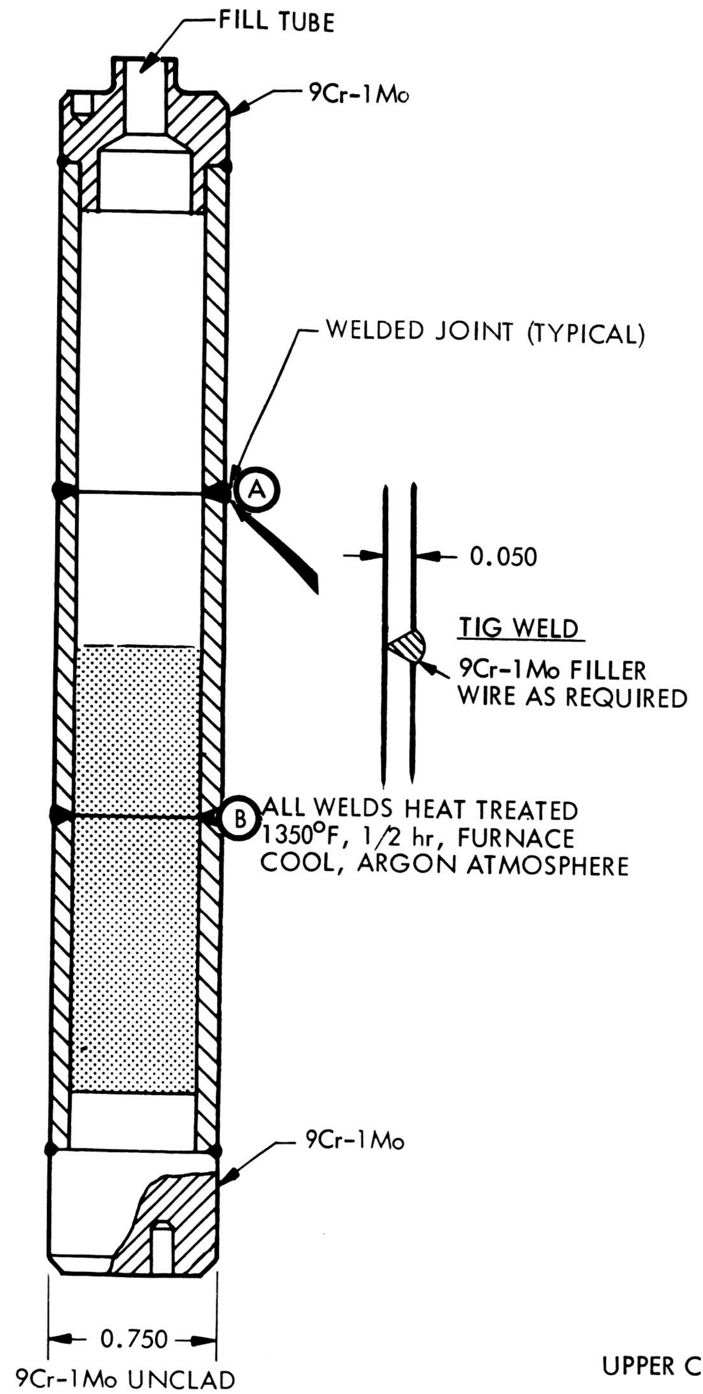
SATISFACTORY UPPER WELD; AFTER 1000 HOUR EXPOSURE

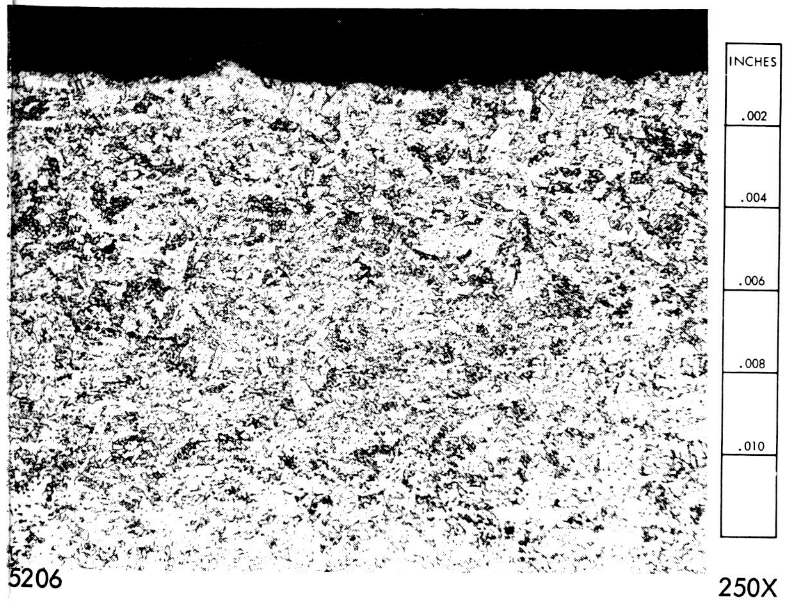
SATISFACTORY LOWER WELD; AFTER 1000 HOUR EXPOSURE

LOWER WELD SHOWING BURN THROUGH OF 316 STAINLESS STEEL WELD INTO Cb LINER; AFTER 1000 HOUR EXPOSURE

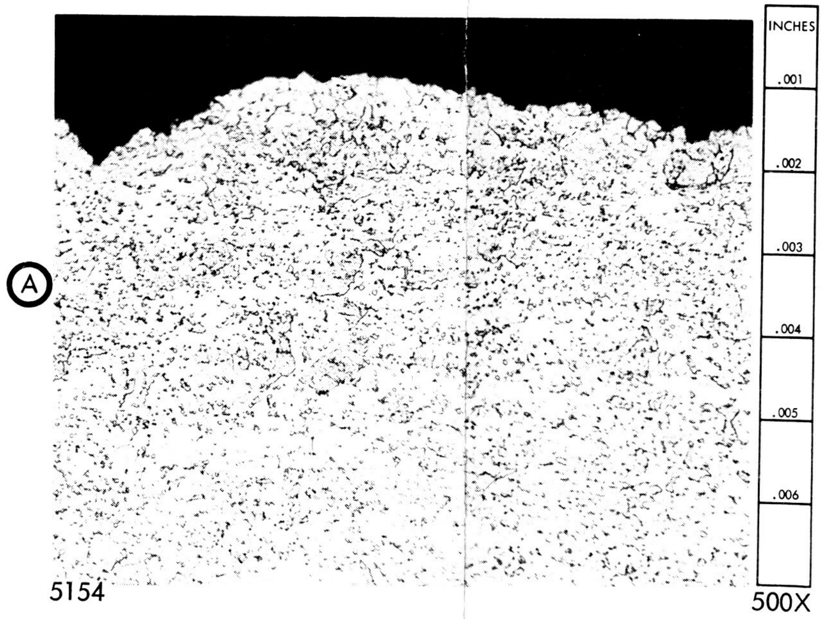
Reflux Capsule, Cb Clad with 316 SS (Test Temperature 1150°F)

Figure 25

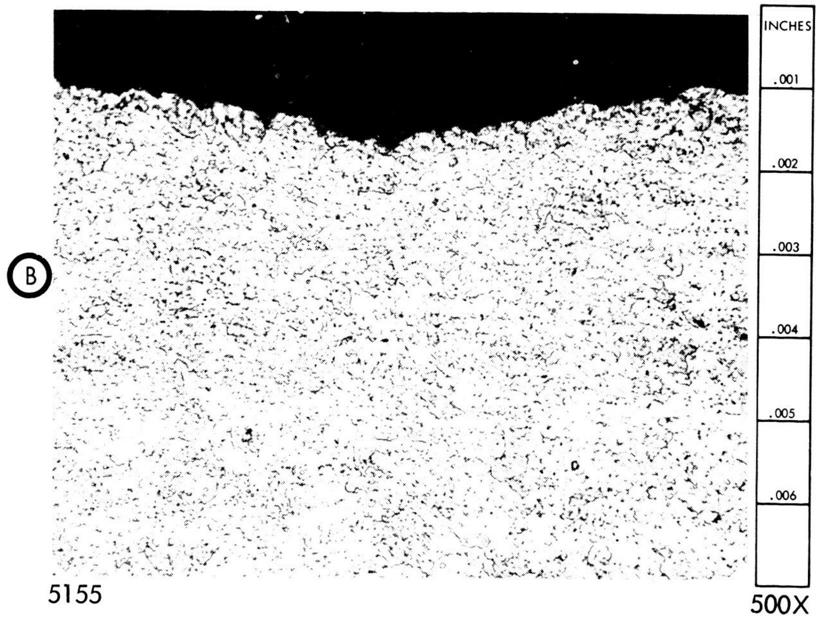

FILL TUBE
9Cr-1Mo
WELDED JOINT (TYPICAL)
A
0.050
TIG WELD
9Cr-1Mo FILLER WIRE AS REQUIRED
B
ALL WELDS HEAT TREATED 1350°F, 1/2 hr, FURNACE COOL, ARGON ATMOSPHERE
9Cr-1Mo
0.750
9Cr-1Mo UNCLAD


INCHES
.002
.004
.006
.008
.010
5206
250X

AS WELDED - 9Cr - 1Mo WELD ZONE
UNEXPOSED


A
INCHES
.001
.002
.003
.004
.005
.006
5154
500X

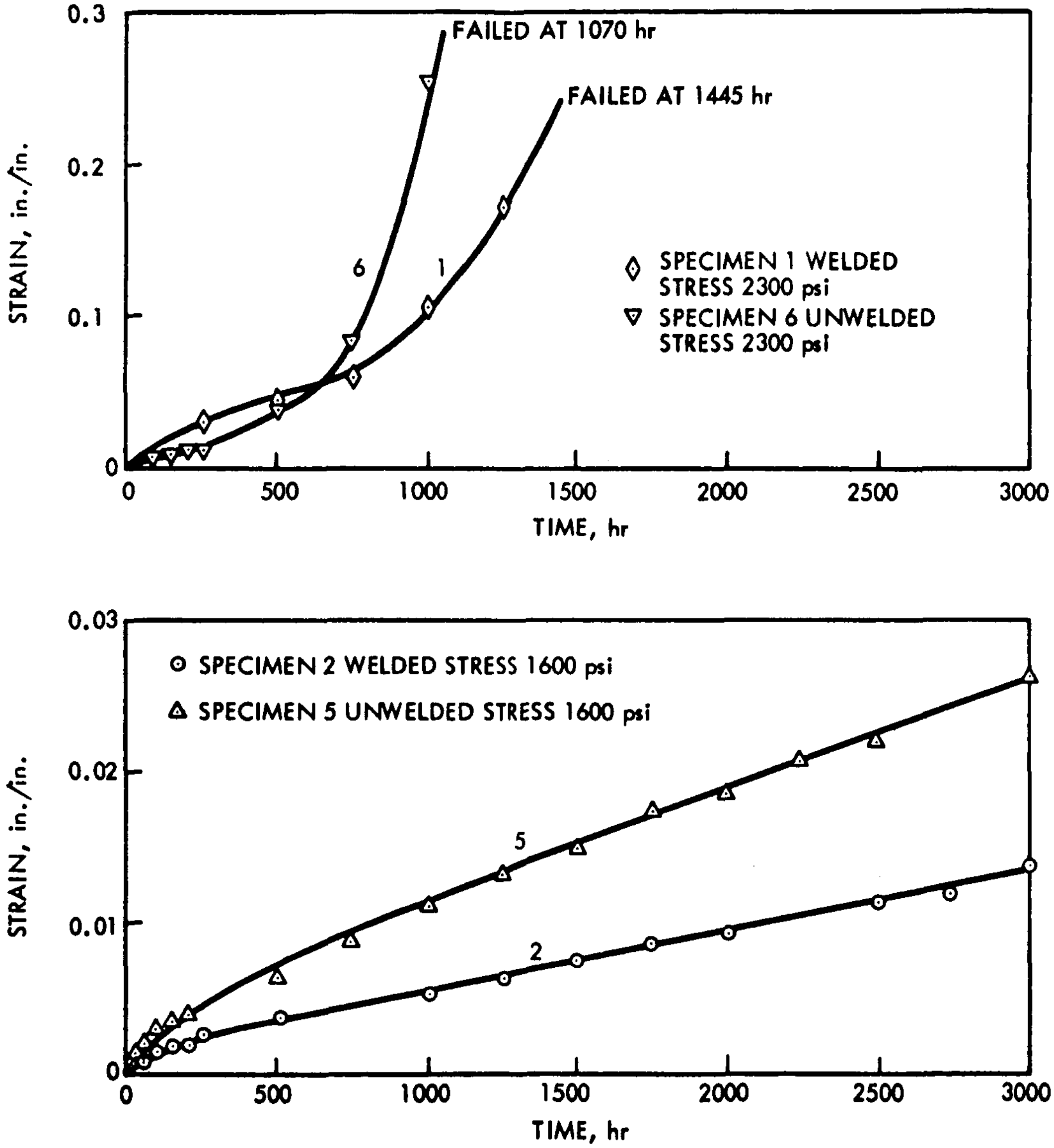
UPPER CAPSULE WELD - 9Cr - 1Mo WELD ZONE; AFTER 1000 HOUR EXPOSURE


B
INCHES
.001
.002
.003
.004
.005
.006
5155
500X

LOWER CAPSULE WELD - 9Cr - 1Mo WELD ZONE; AFTER 1000 HOUR EXPOSURE

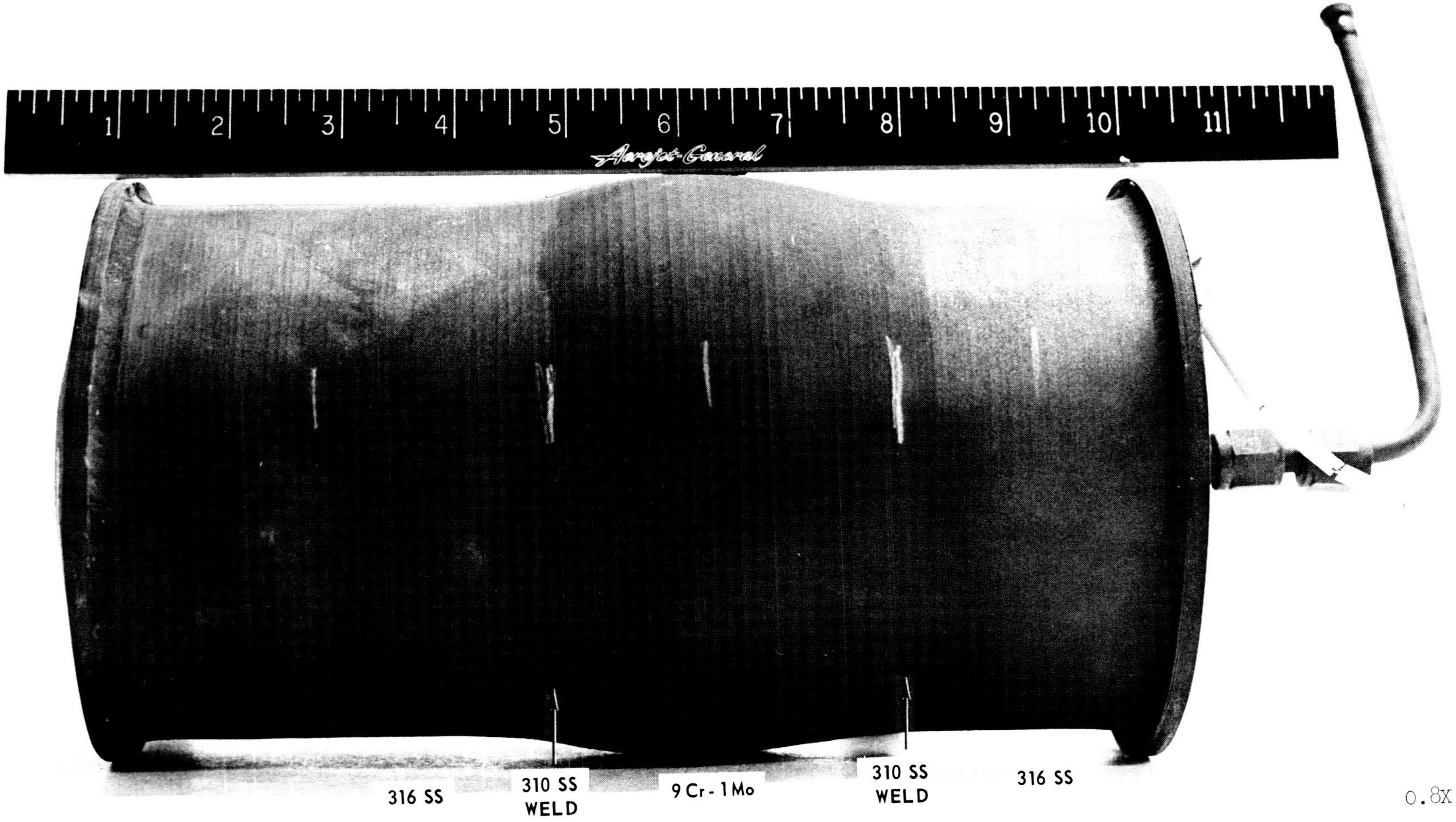
Reflux Capsule, 9Cr-1Mo Unclad (Test Temperature 1150°F

Figure 26



Creep Curves, 9Cr-1Mo Welded and Unwelded Specimens
(Tested at 1325°F in Air)

Figure 27

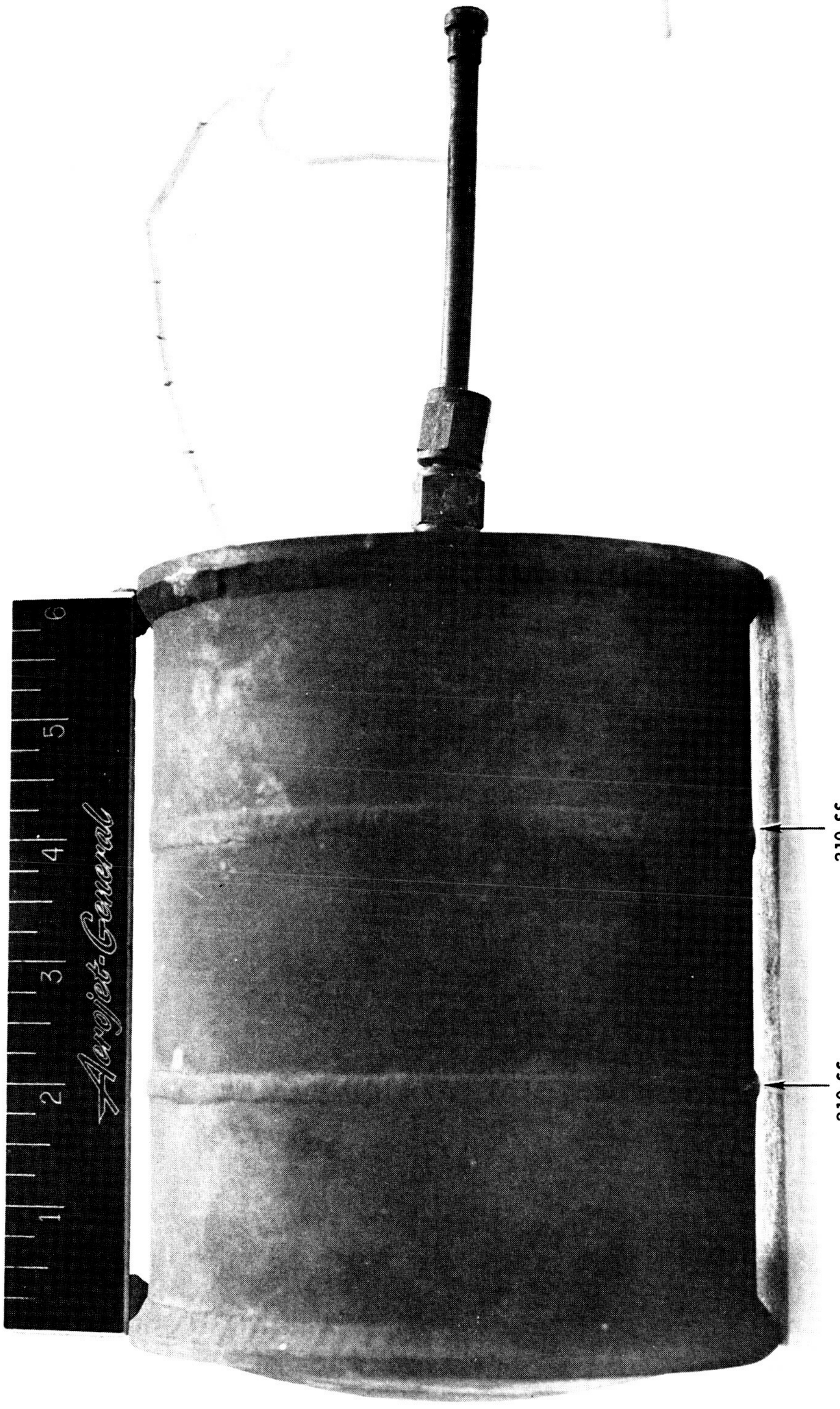

1
2
3
4
5
6
7
8
9
10
11
Aerojet-General
316 SS
310 SS WELD
9 Cr-1 Mo
310 SS WELD
316 SS

0.8X

9Cr-1Mo/316 SS Transition Joint Test Specimen (Welded and Roll Planished) After 2402 Hours at 1300°F; Wall Hoop Stress - 1470 psi

Figure 28

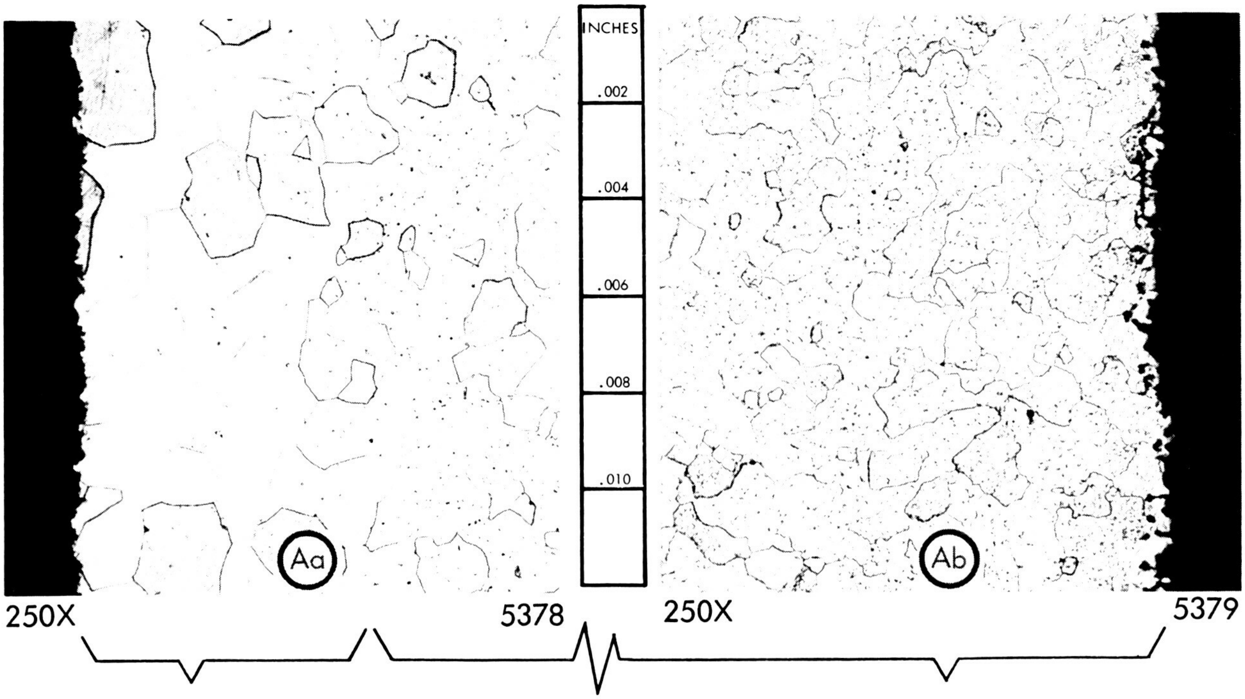
0.8X


316 SS
310 SS WELD
9 Cr-1 Mo
310 SS WELD
316 SS
1
2
3
4
5
6
Aerojet-General

9Cr-1Mo/316 SS Transition Joint Test Specimen (Welded) After 2402 Hours at 1300°F; Wall Hoop Stress - 1100 psi

864 696

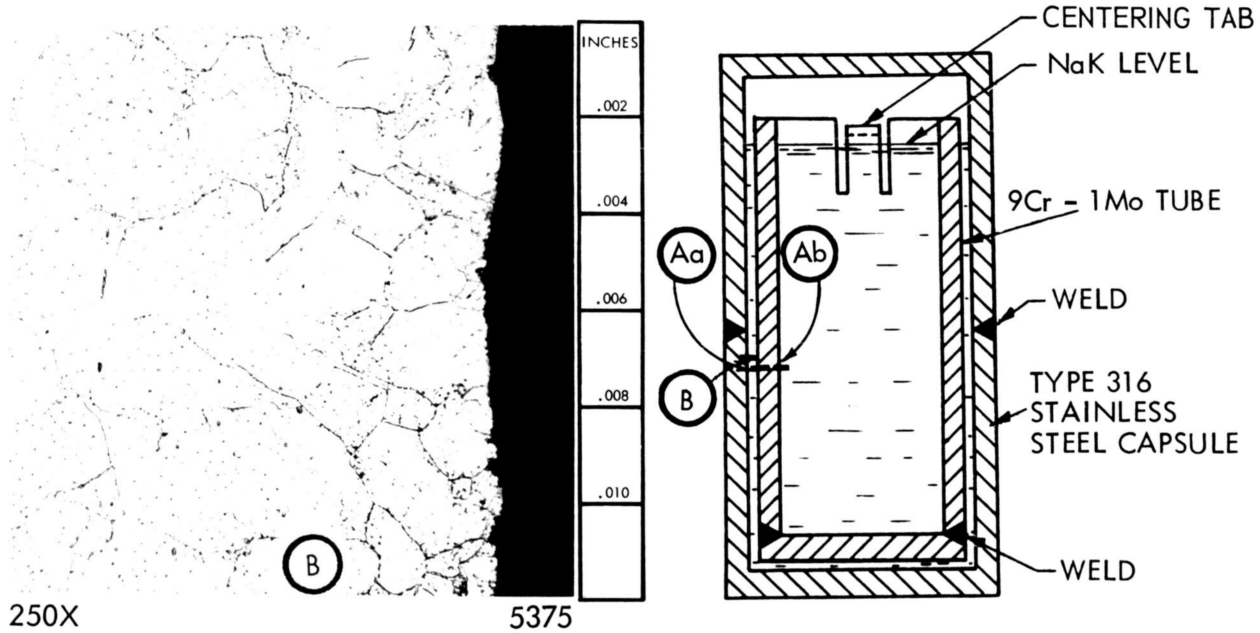
Figure 29


INCHES
.002
.004
.006
.008
.010
Aa
Ab
250X
5378
250X
5379

DECARBURIZED
GRAIN SIZE: ASTM 5

NOT DECARBURIZED
GRAIN SIZE: ASTM 6 1/2

a. 9Cr - 1Mo TUBE - GLYCEREGIA ETCH


INCHES
.002
.004
.006
.008
.010
B
250X
5375
CENTERING TAB
NaK LEVEL
9Cr - 1Mo TUBE
Aa
Ab
B
WELD
TYPE 316
STAINLESS
STEEL CAPSULE
WELD

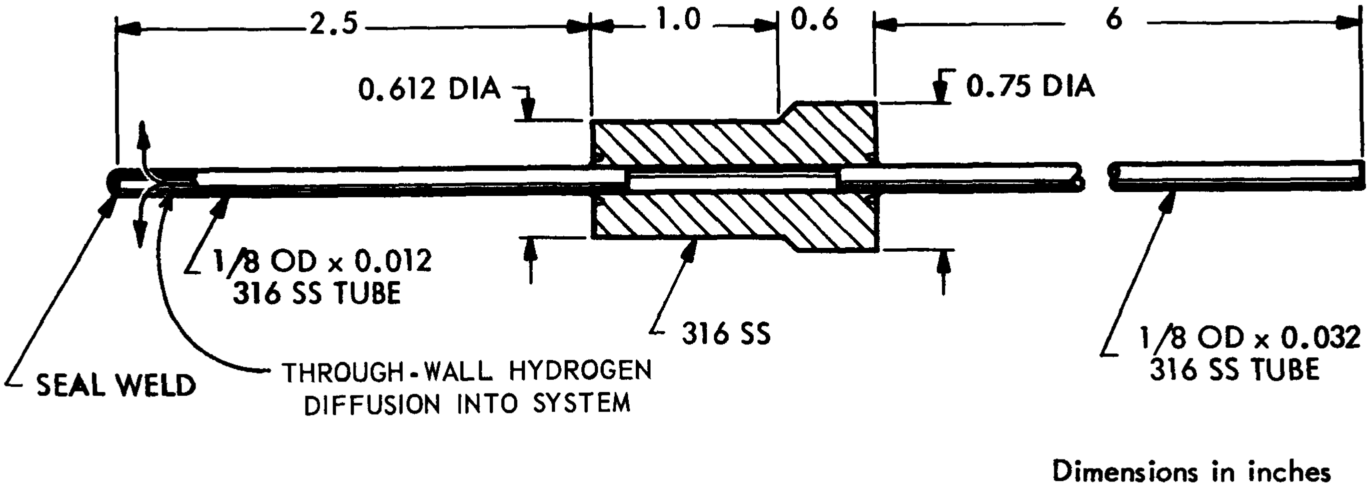
NOT APPRECIABLY CARBURIZED

b. TYPE 316 STAINLESS STEEL
TUBE, GLYCEREGIA ETCH

c. CROSS SECTION OF
CAPSULE

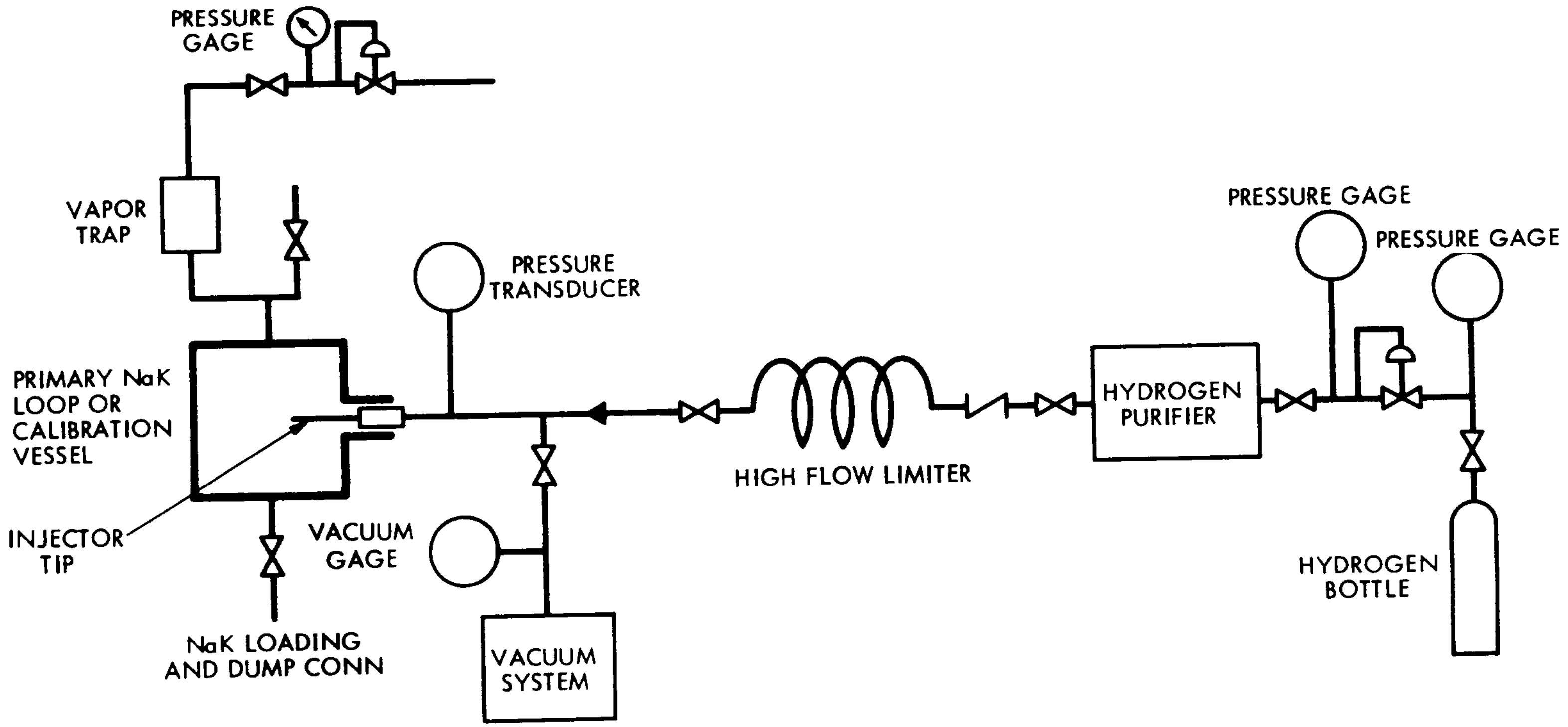
Microstructure of Trial Capsule After 500 Hours at 1325°F in NaK

Figure 30


2.5
1.0
0.6
6
0.612 DIA
0.75 DIA
1/8 OD x 0.012
316 SS TUBE
316 SS
1/8 OD x 0.032
316 SS TUBE
SEAL WELD
THROUGH-WALL HYDROGEN
DIFFUSION INTO SYSTEM
Dimensions in inches

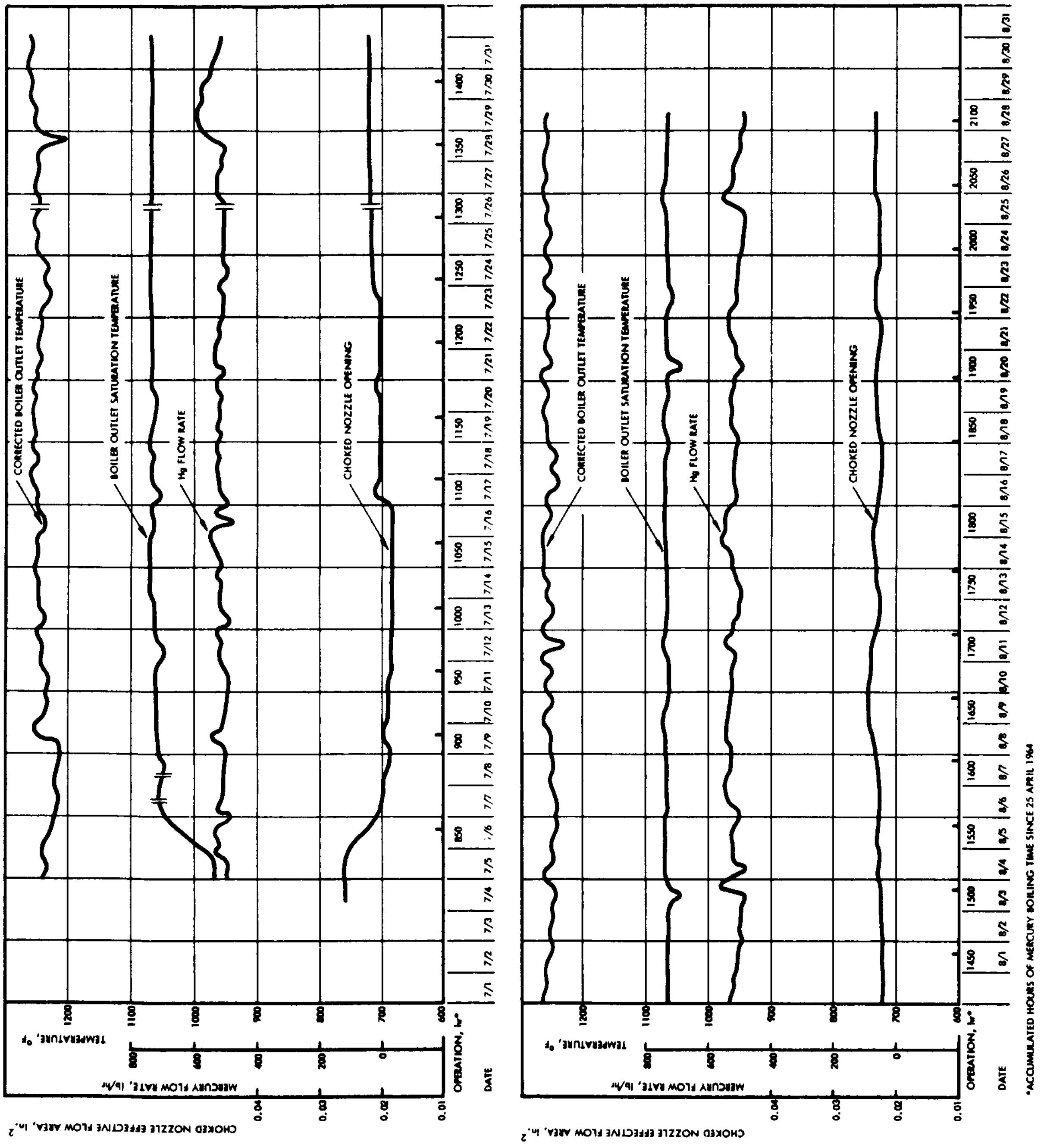
Hydrogen Injector Tip

Figure 31


PRESSURE GAGE
VAPOR TRAP
PRIMARY NaK LOOP OR CALIBRATION VESSEL
INJECTOR TIP
NaK LOADING AND DUMP CONN
PRESSURE TRANSDUCER
VACUUM GAGE
VACUUM SYSTEM
HIGH FLOW LIMITER
HYDROGEN PURIFIER
PRESSURE GAGE
PRESSURE GAGE
HYDROGEN BOTTLE

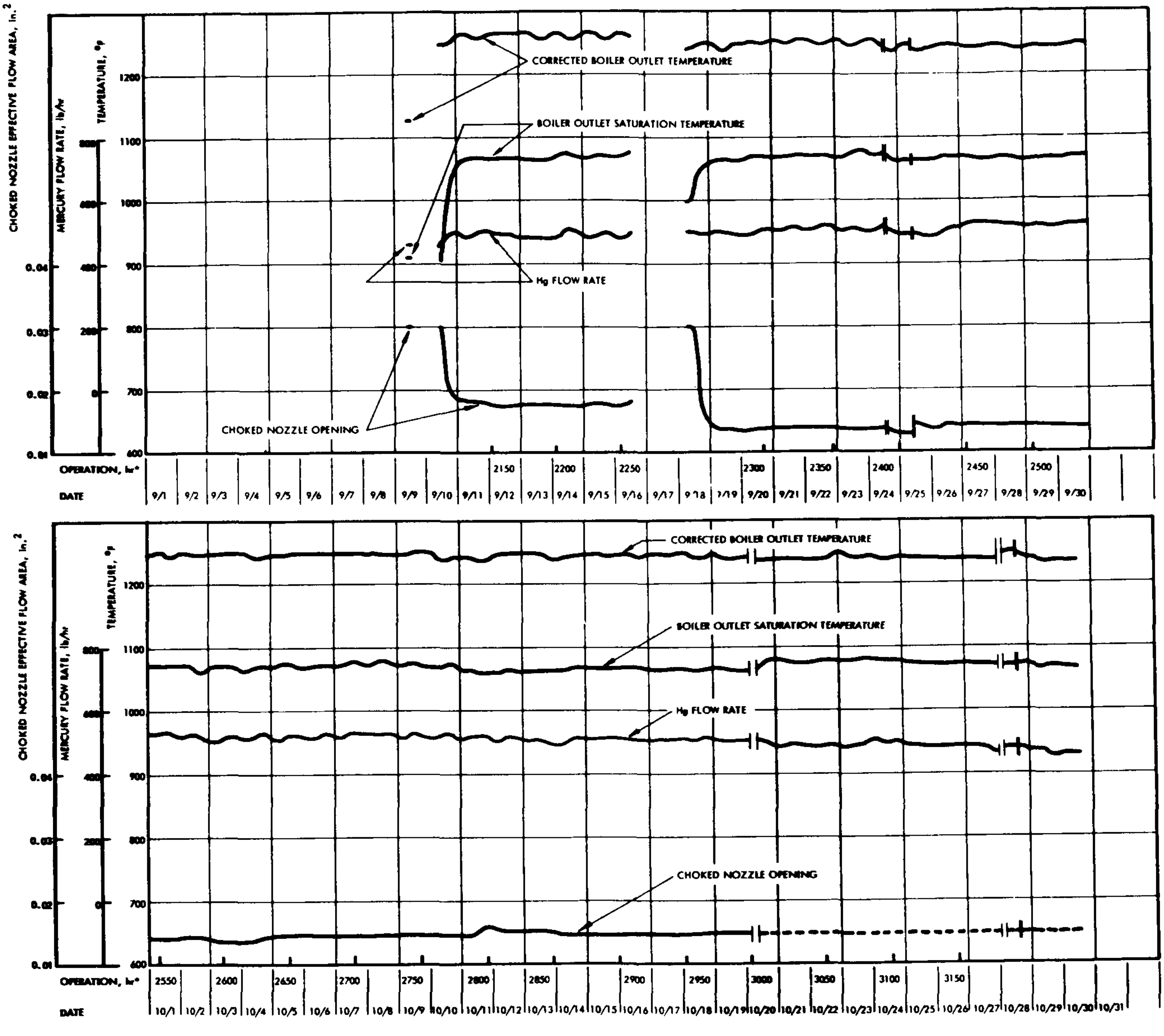
Hydrogen Injection System Flow Diagram

Figure 32



Operating Data for CL-3

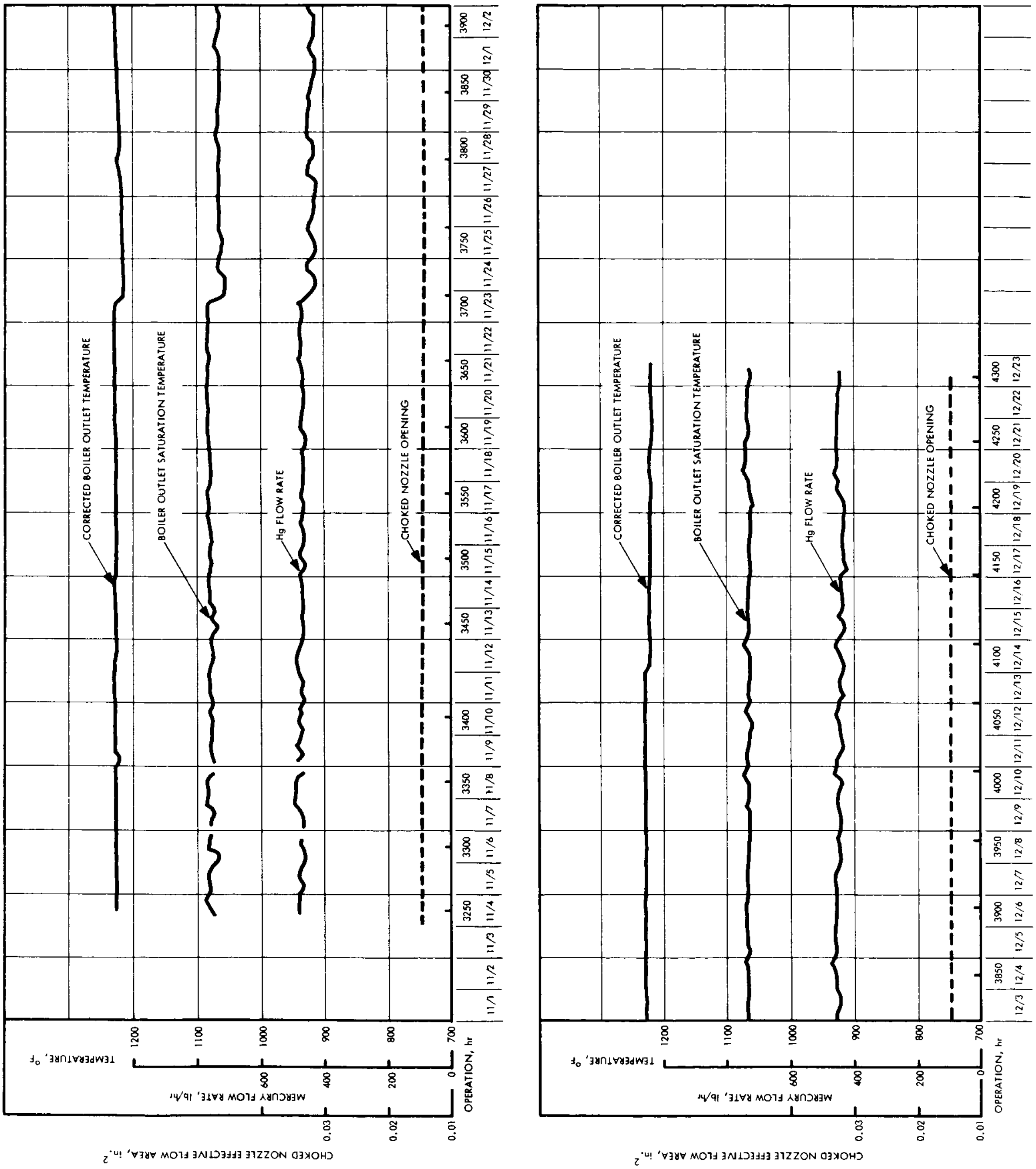
Figure 33a



* ACCUMULATED HOURS OF MERCURY BOILING TIME SINCE 25 APRIL 1964

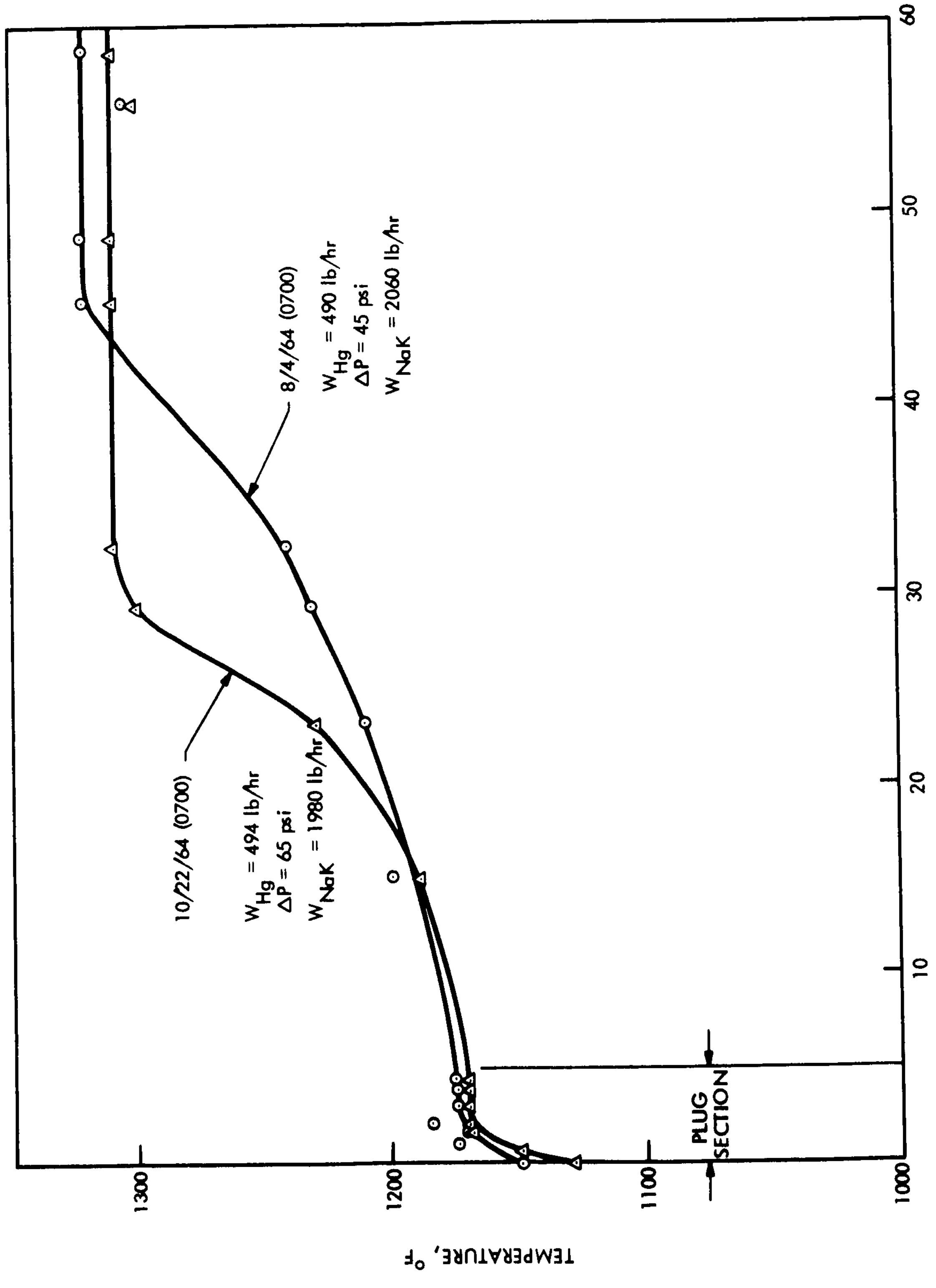
Operating Data for CL-3

Figure 33b



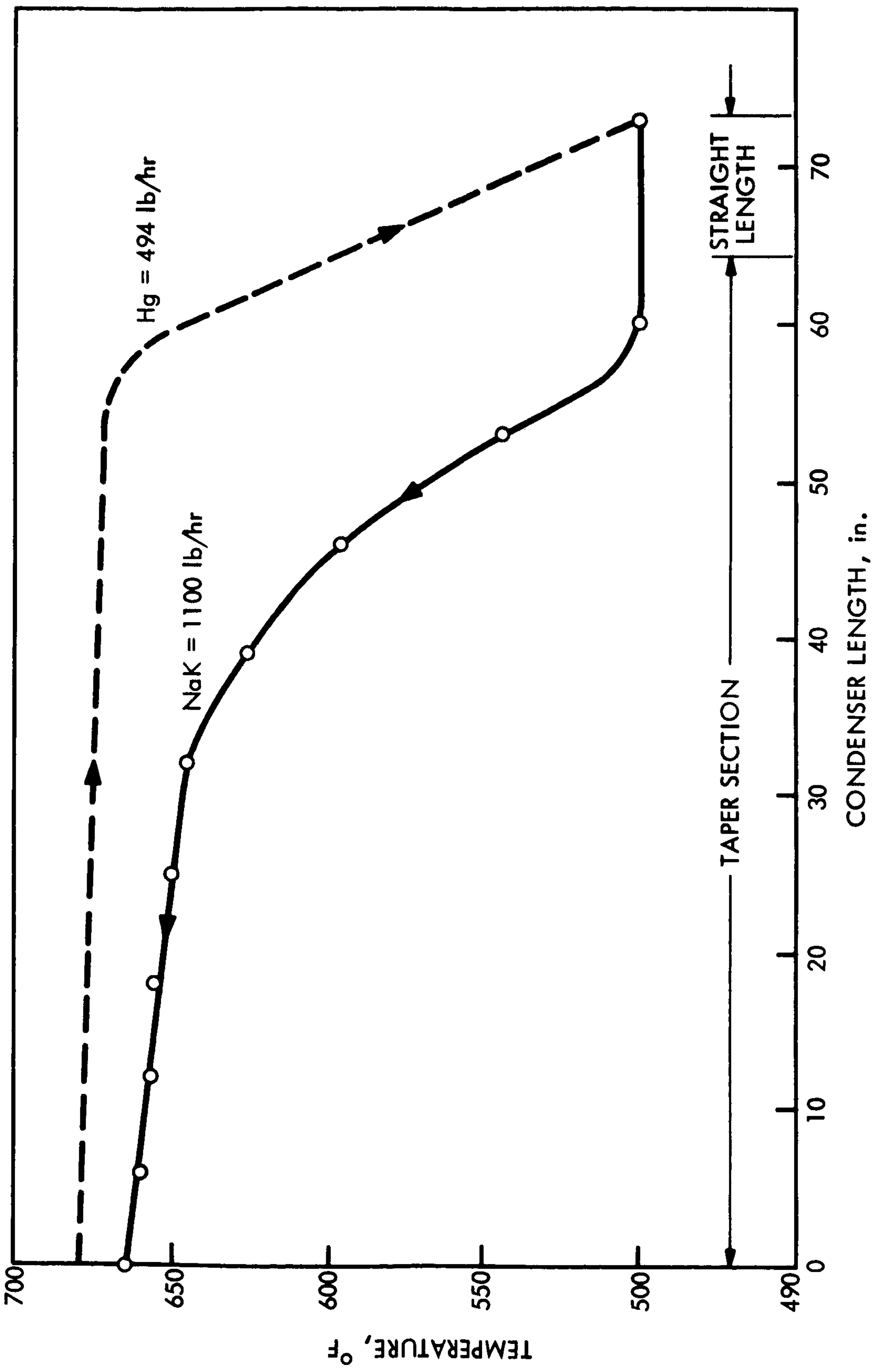
Operating Data for CL-3

Figure 33c



Mercury Boiler, Temperature Profile

Figure 34



Mercury Condenser, Temperature Profile

Figure 35

Report No. 2989

NASA CONTRACTOR REPORT

# SNAP-8 MATERIALS REPORT FOR JULY-DECEMBER 1964

Aerojet-General Corporation

## ABSTRACT

Investigations of materials were initiated, or continued, in areas where the established state of the art was not well enough developed to permit a reliable material selection, and to assist in the design, development, fabrication, and testing of various SNAP-8 components.

A mercury forced-convection corrosion-loop investigation was continued with the aim of evaluating the corrosion resistance of 9Cr-1Mo alloy steel for 10,000-hour service. Work was also initiated or continued, on evaluation of the resistance to mercury corrosion of candidate backup mercury-containment materials.

Data were developed to substantiate the selection of 9Cr-1Mo steel as the mercury-containment material for the SNAP-8 system, and to evaluate potential problems related to its use.

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